University of Victoria

Recent STEHM High-Resolution Performance and Future Applications

CAMTEC Workshop
July 5, 2013

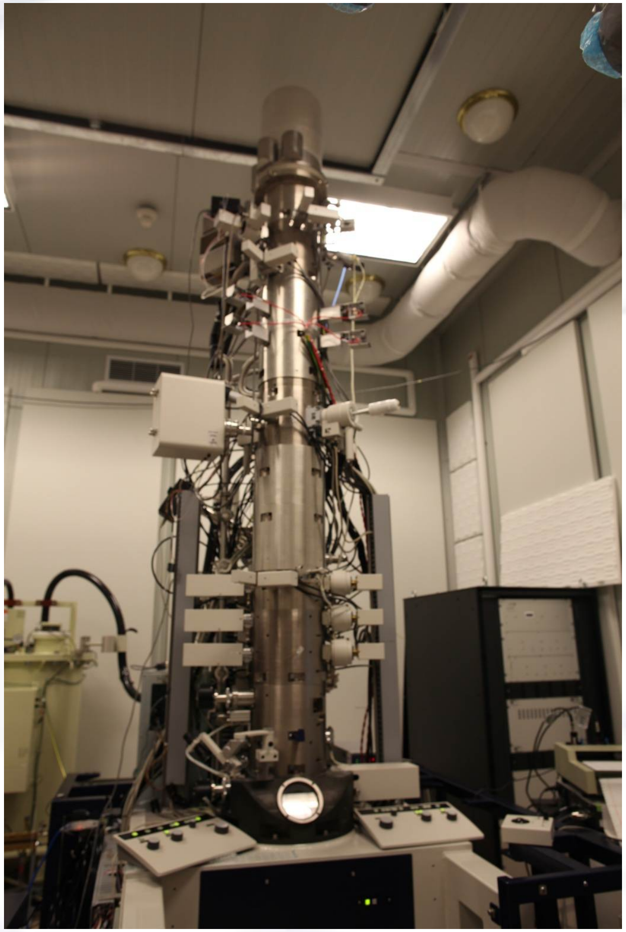
Rodney Herring
University of Victoria,
Victoria, BC Canada
V8W 2Y2 (rherring@uvic.ca)

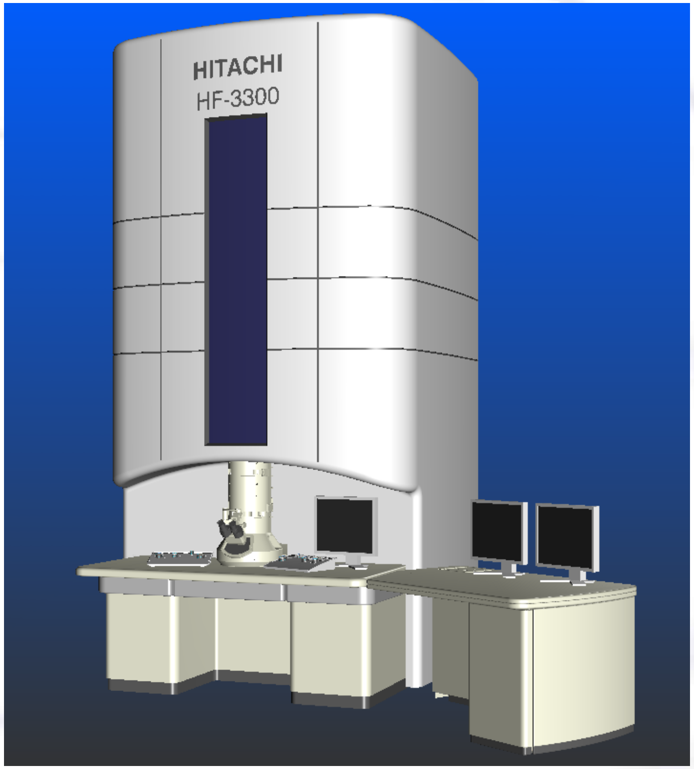
Recent STEHM High-Resolution Performance and Future Applications

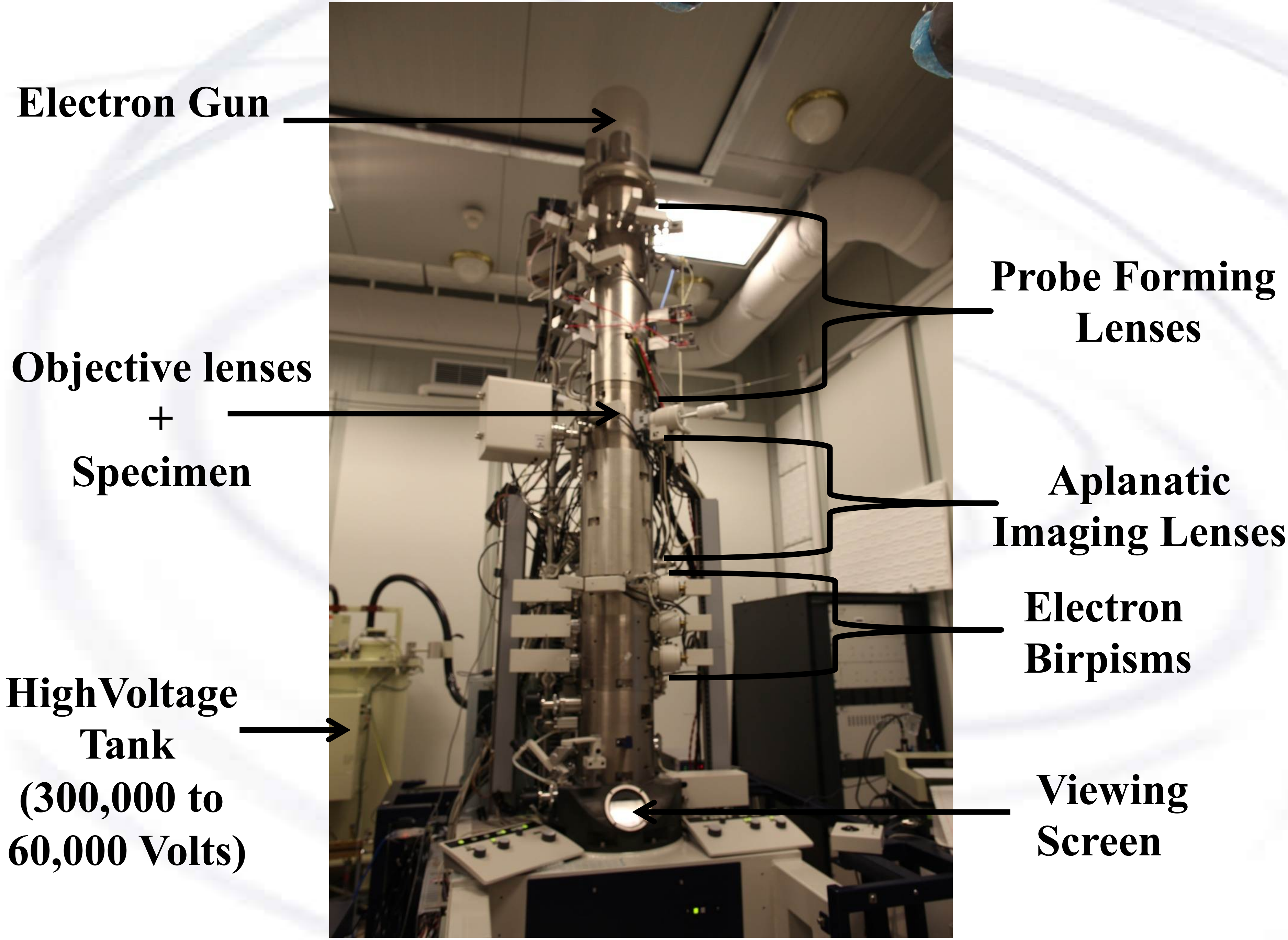
Three presentations

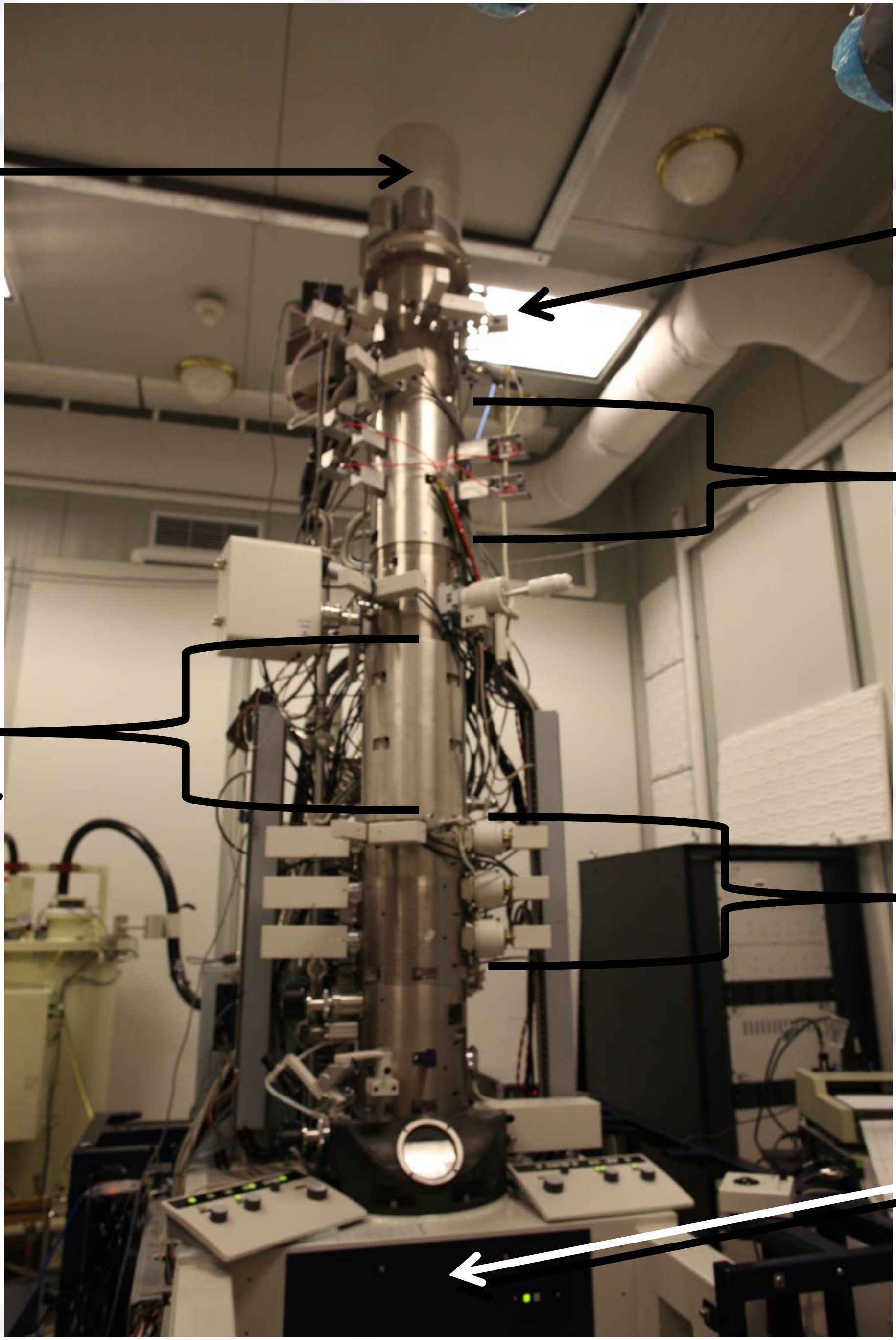
- Structure of the STEHM plus recent performance and possible applications
- Special features of STEHM plus possible applications
- Cost of STEHM

The STEHM

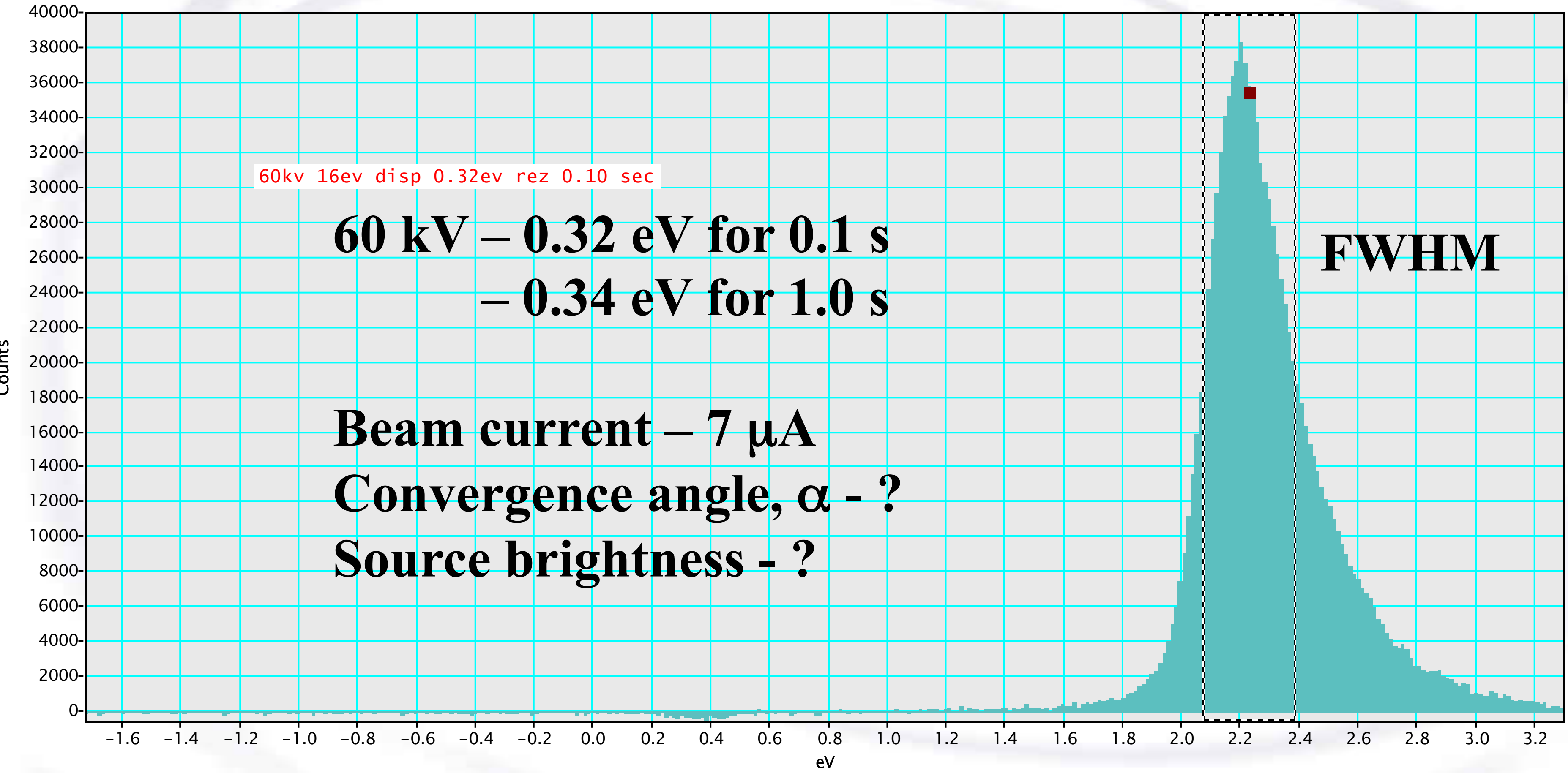





Electron Gun
Probe Forming Lenses
Objective lenses + Specimen
Aplanatic Imaging Lenses
Electron Birpisms
HighVoltage Tank (300,000 to 60,000 Volts)
Viewing Screen


Cold-field Electron Source (10^{-13} torr)
1 electron biprism + dislocated hologram aperture
STEM Cs + Cc aberration corrector (ExB Wien filter) (CEOS SC-COR)
Aplanatic TEM Cs + Coma aberration corrector (CEOS B-COR)
3 electron biprisms + extra lenses
EELS + GIF

The STEHM's Energy Spread

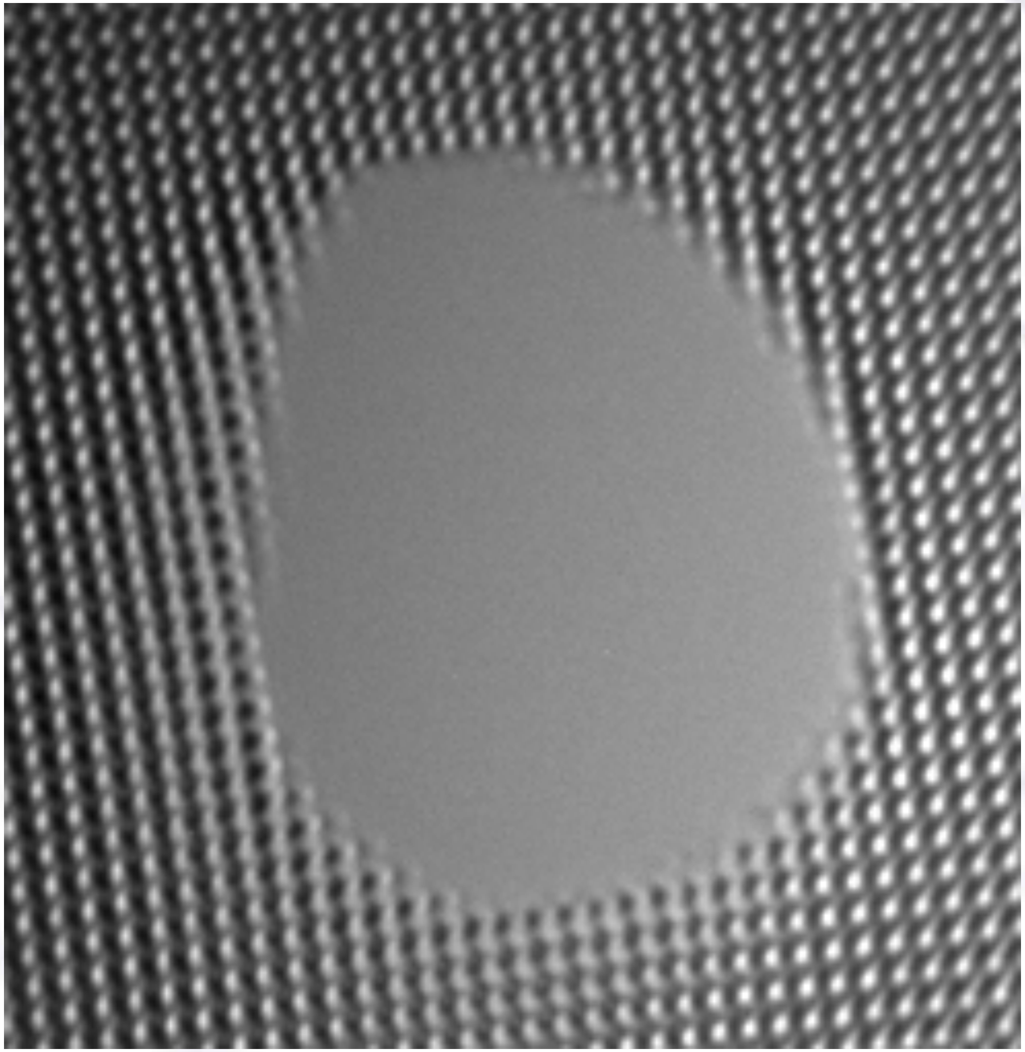


Maximum emission stable for hours – high emission for days.
Performance needs to be measured. Will be reported later.
The energy spread will improve with age as the tip flattens.

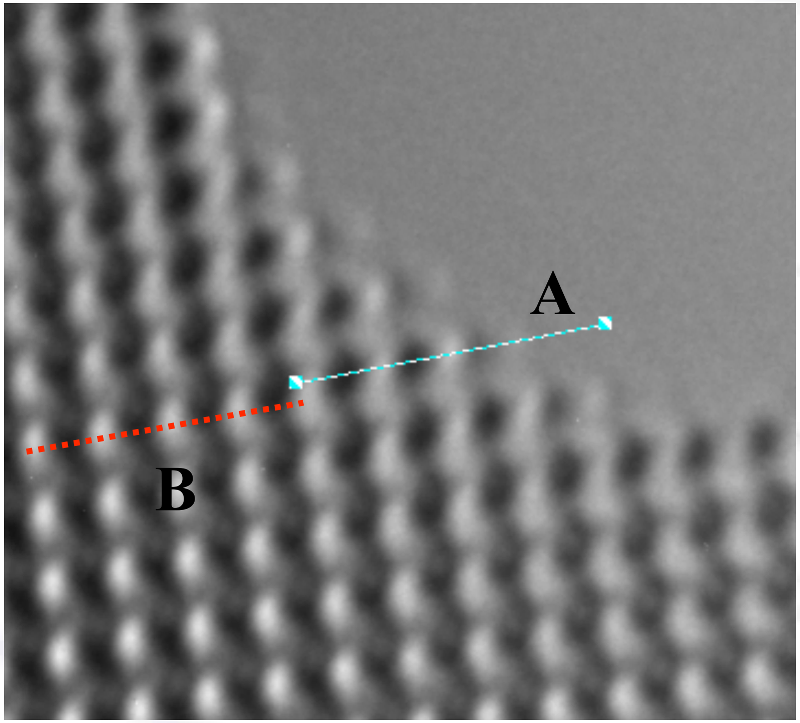
STEHM's TEM

Au lattice image

TEM specimen made insitu by specimen ablation by electron beam



TEM Imaging at Edge of Specimen

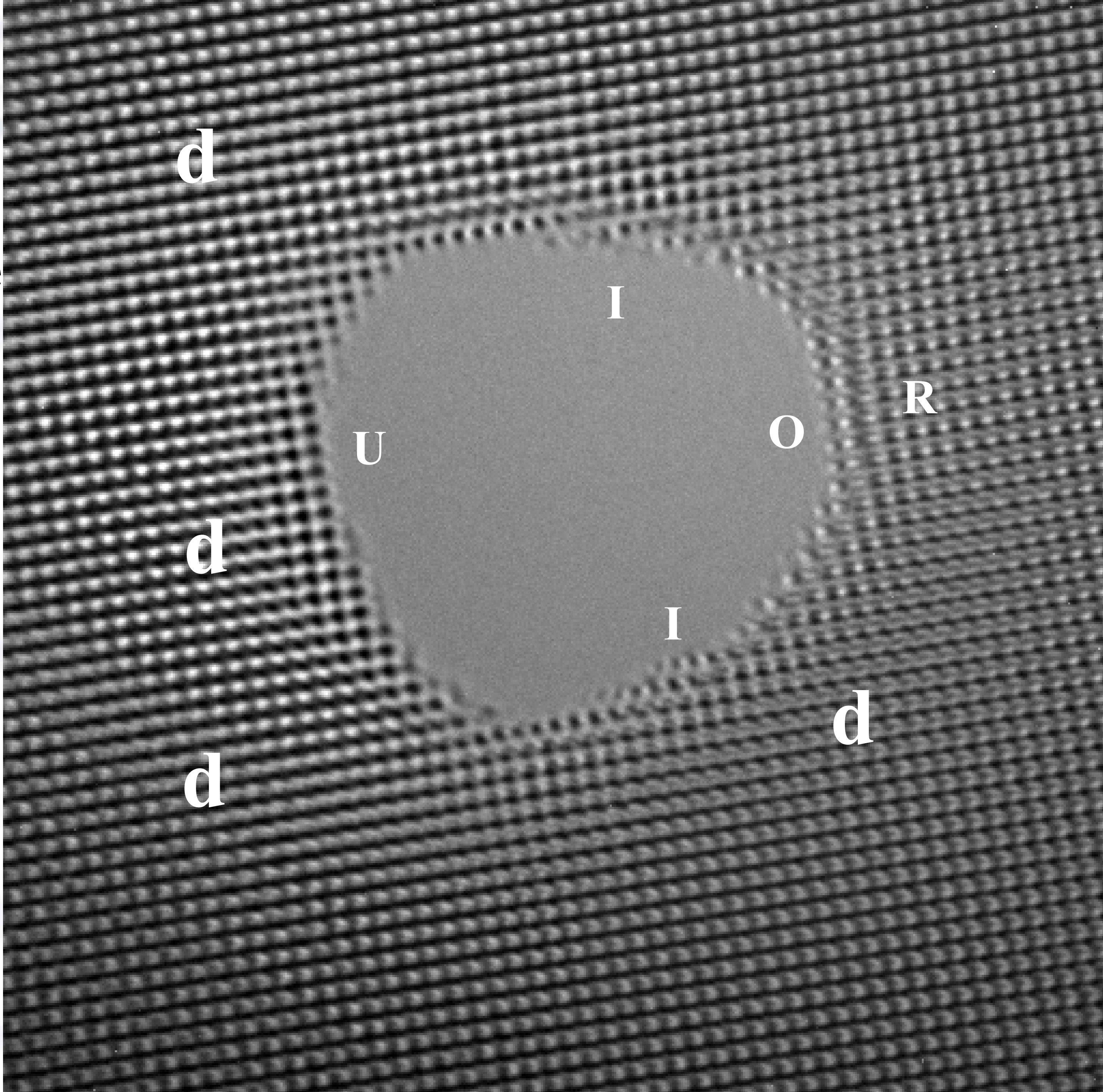


Note the change in channeling of the electrons from passing around the atoms (dark contrast) to along the atomic core (white contrast)

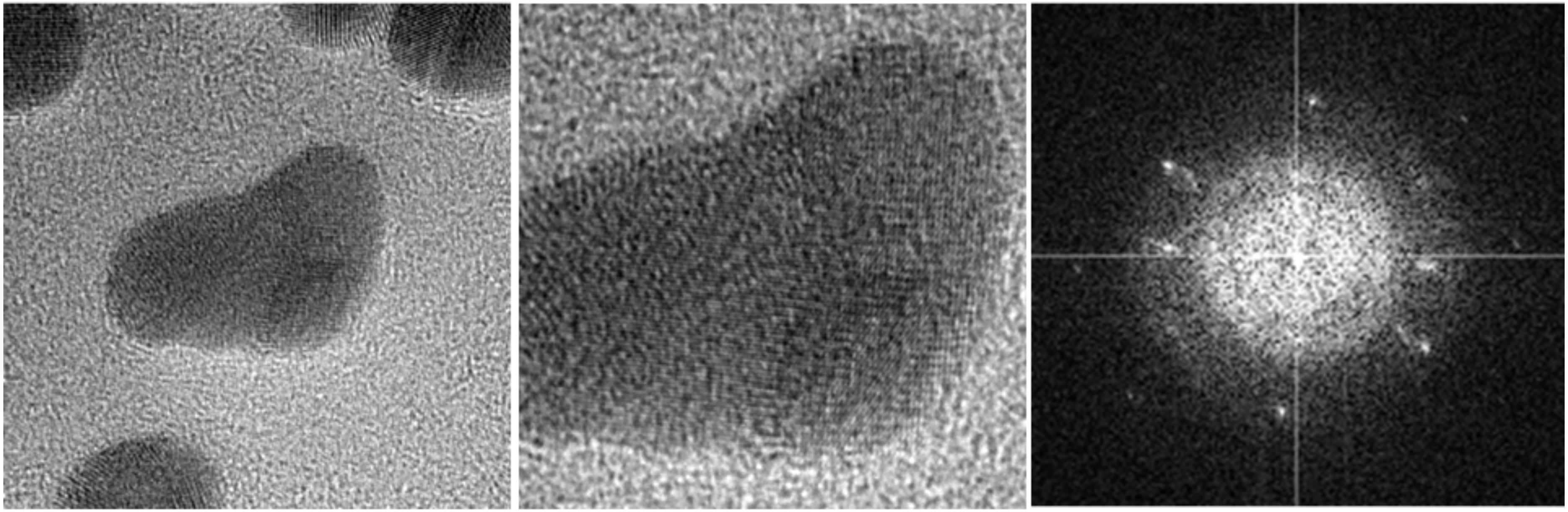
A – electrons traveling around the outside of atomic column
B – electrons traveling along core of atomic column

STEHM's TEM Image

Au lattice image showing dynamic imaging contrast, d, under focus, U, over focus, O, exactly infocus, I, atomic plane relaxation, R.

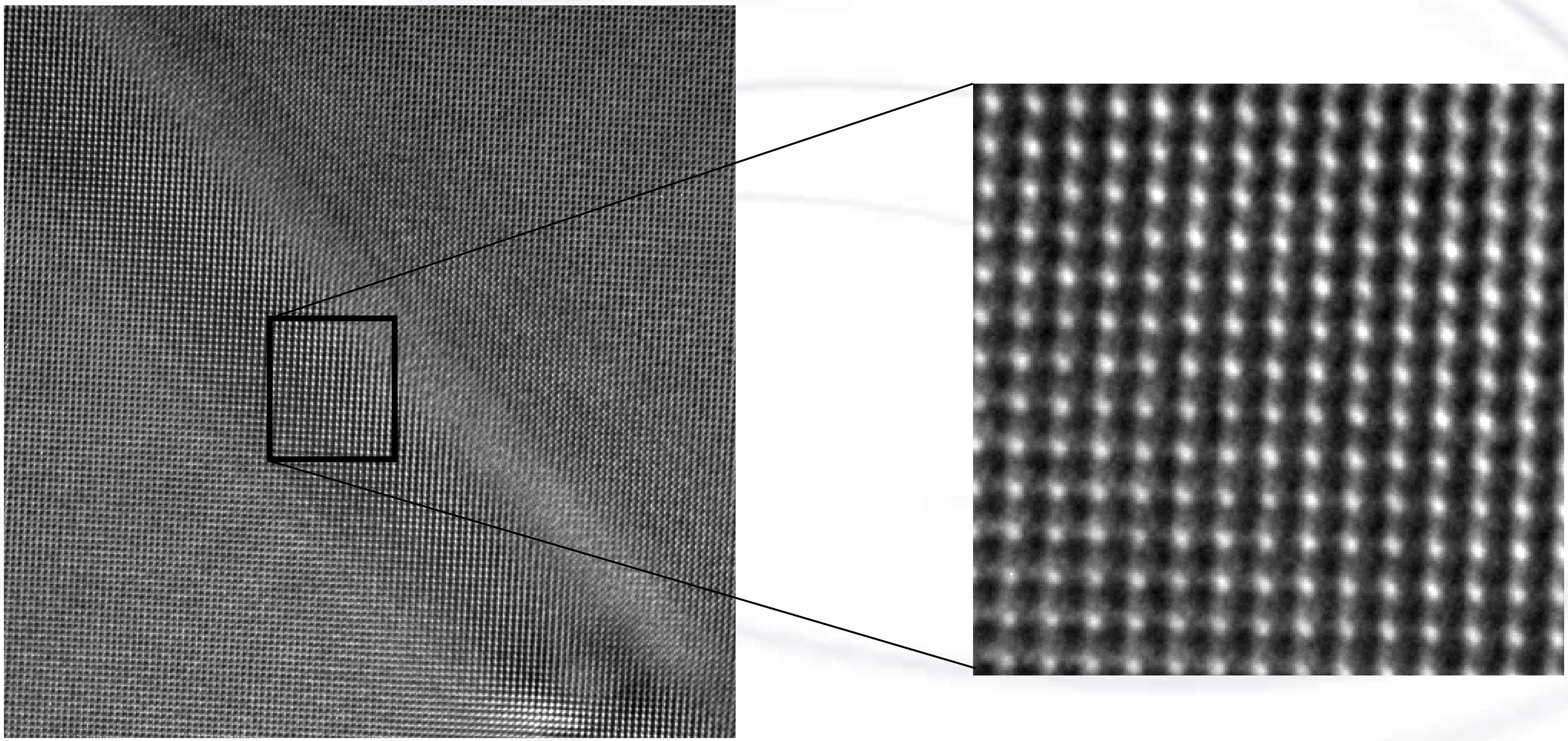


Ultra-stable STEHM Imaging



TEM image of gold crystals on an amorphous carbon substrate taken for 120 seconds, the maximum recording time available, using the Gatan USC 1004 2k x 2k camera, b) enlargement of the centrally located gold crystal to more easily see the presence of lattice fringes, verified in c) the Fourier transform. Useful for low-dose, beam-sensitive specimen such as biology and soft specimen.

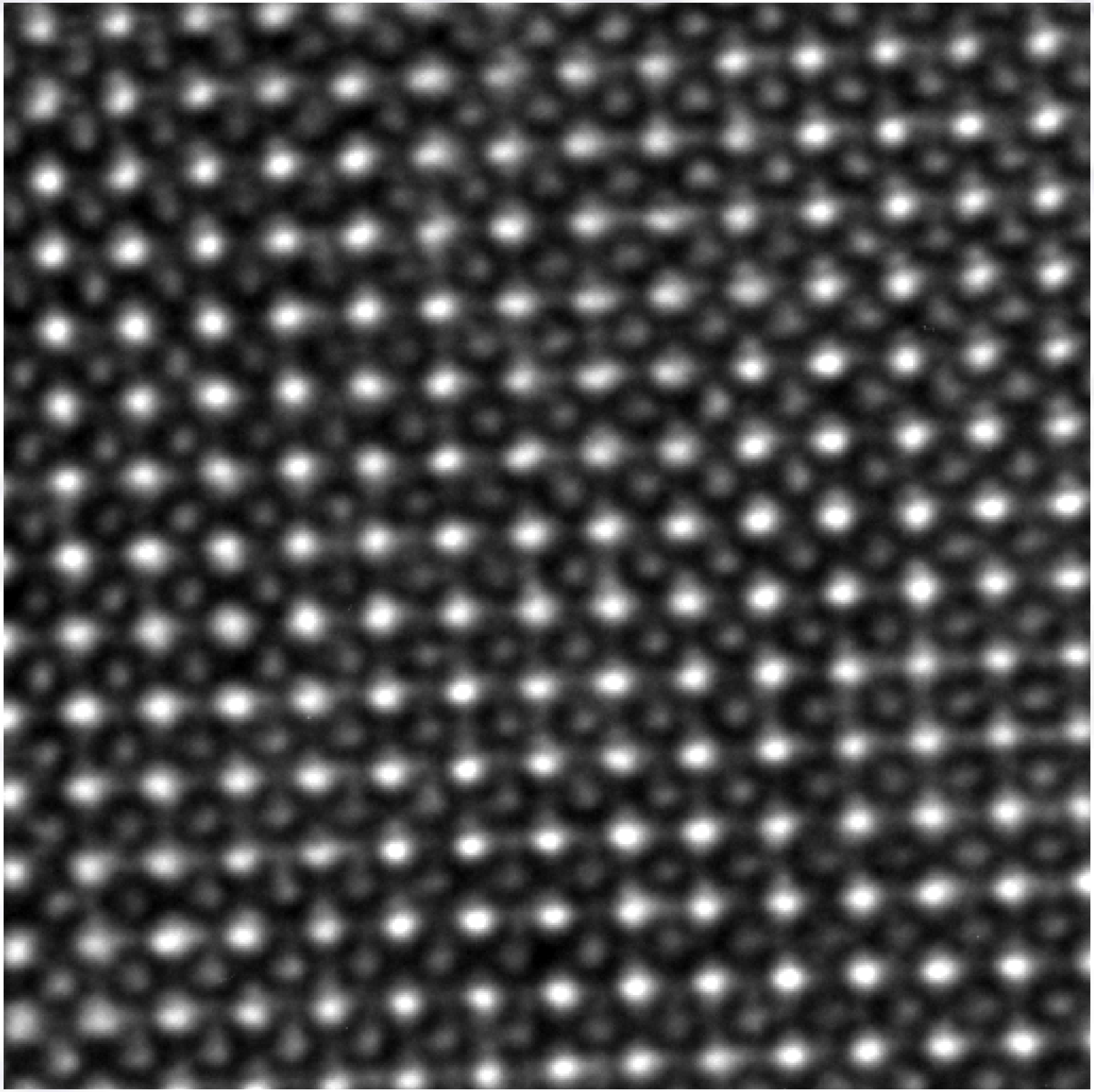
STEHM's TEM Au Lattice Imaging



Atomic core imaging condition

STEHM imaging Of $BaTiO_3$

Atomic core imaging



Before arriving at UVic

STEHM's TEM

Coherent Information Transfer

measured by Fourier Transform

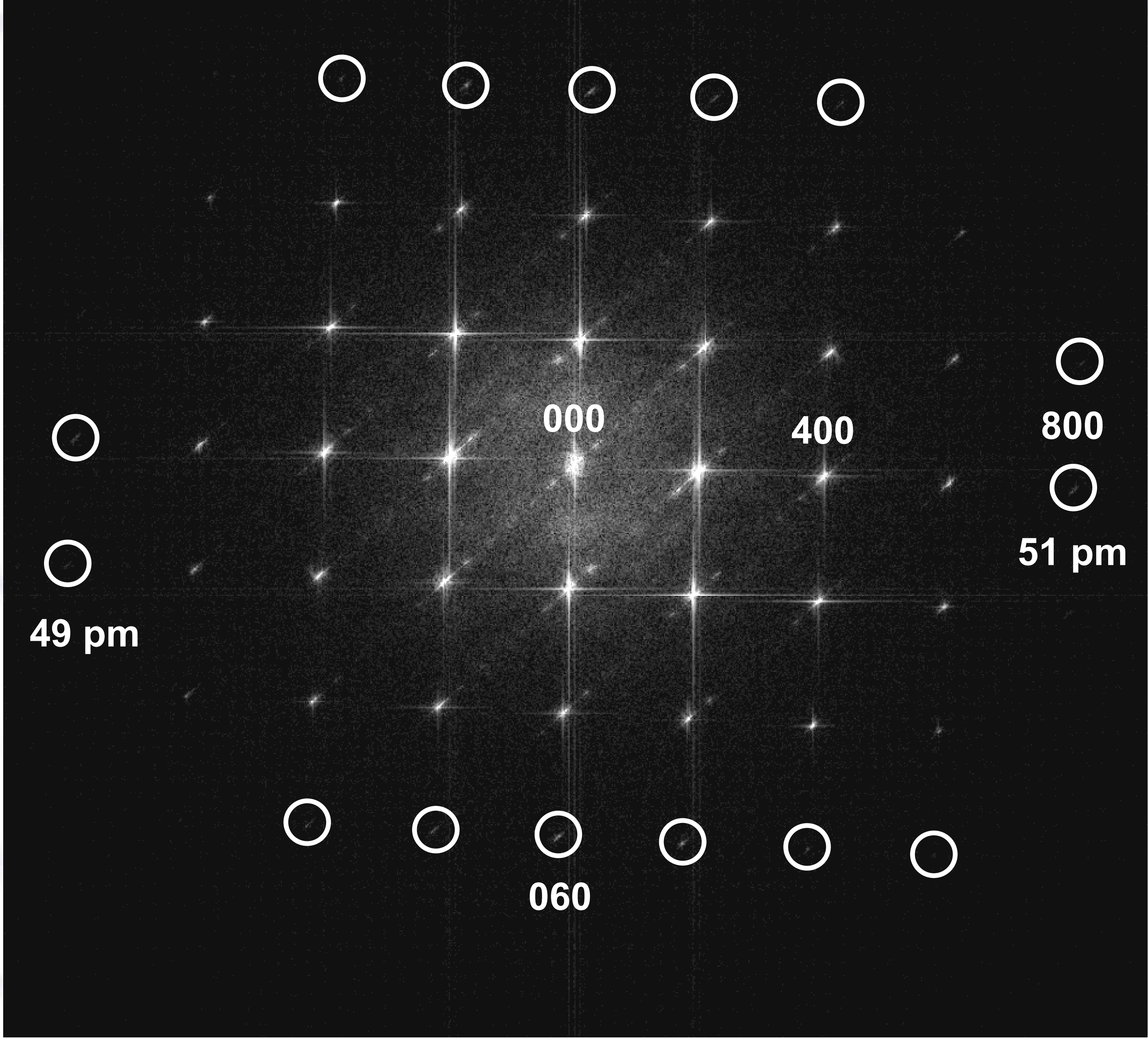
Lattice Parameter of Au

$$a_o = 4.08\ \text{Å}$$

using

$$[010]$$

zone axis



STEHM's TEM

Coherent Information Transfer

measured by Fourier Transform

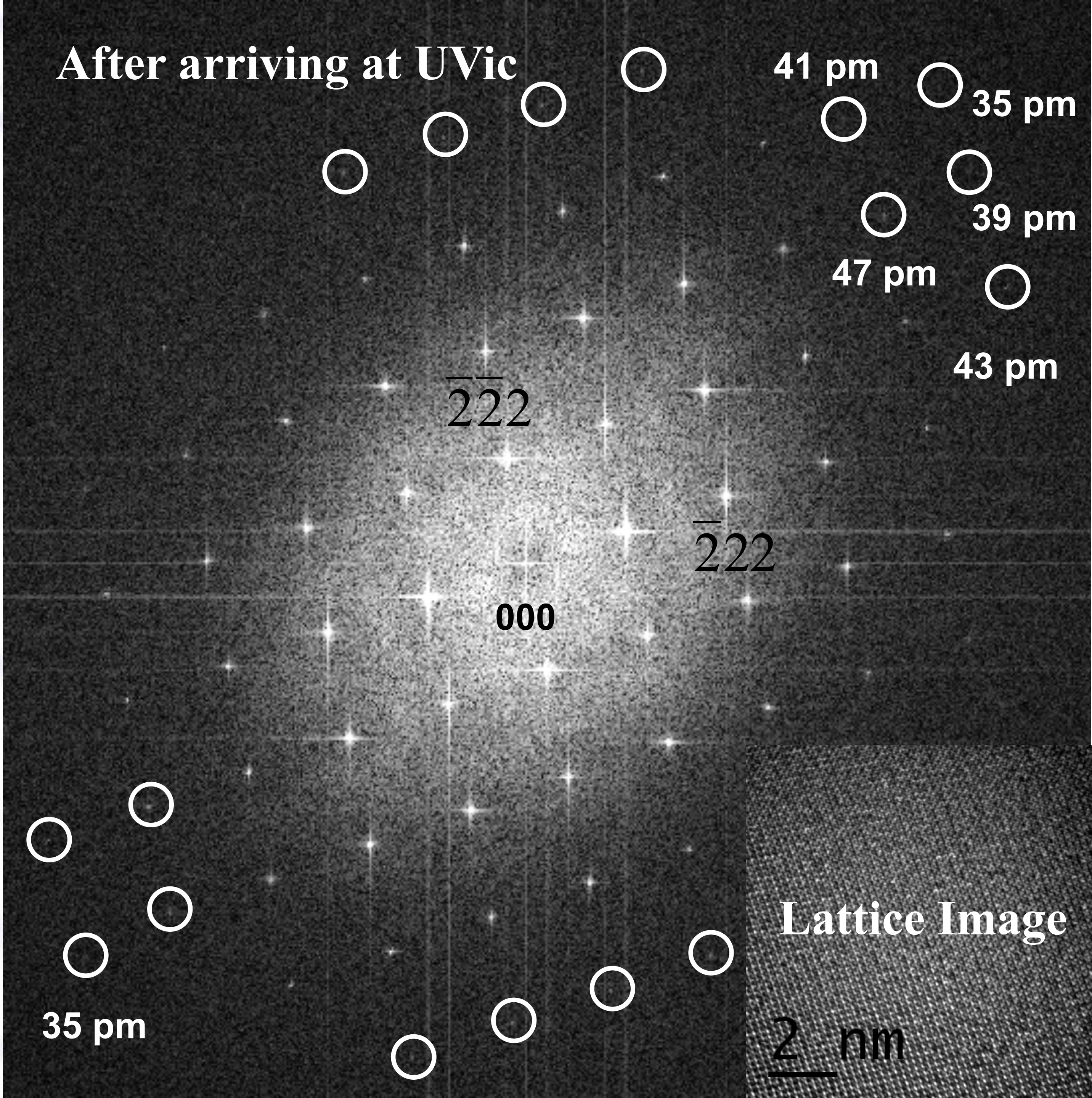
Lattice Parameter of Au

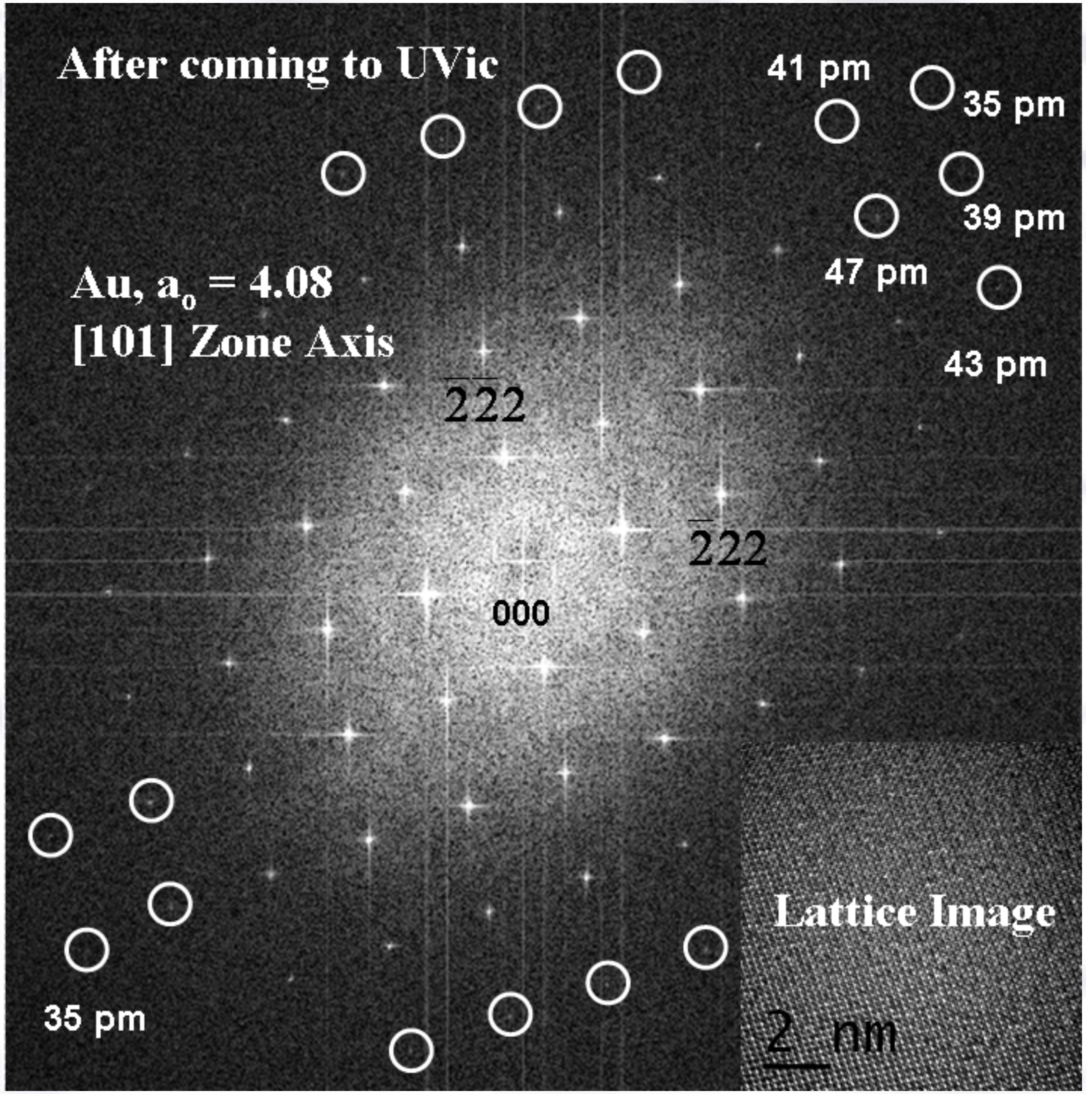
$a_o = 4.08\ \dot{A}$

using

$[101]$

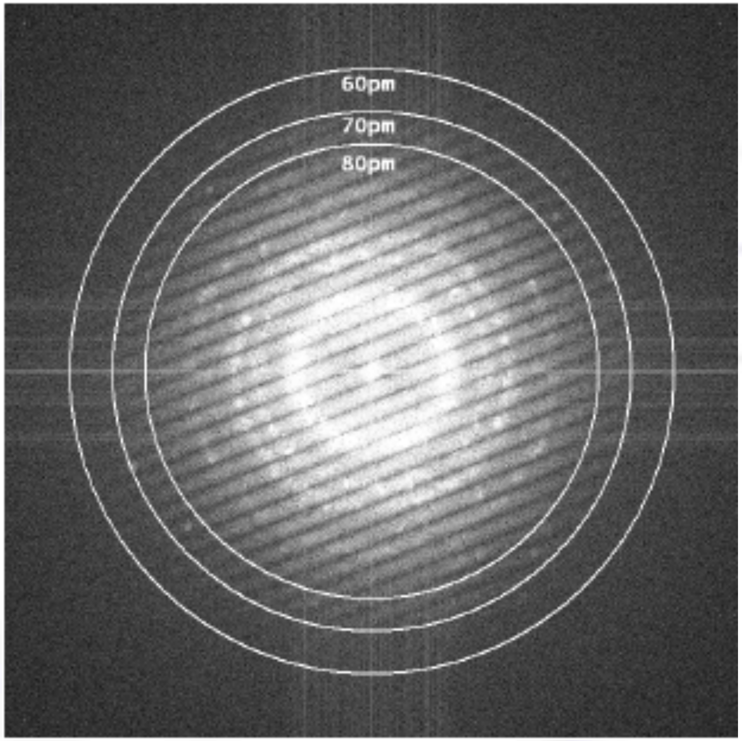
zone axis



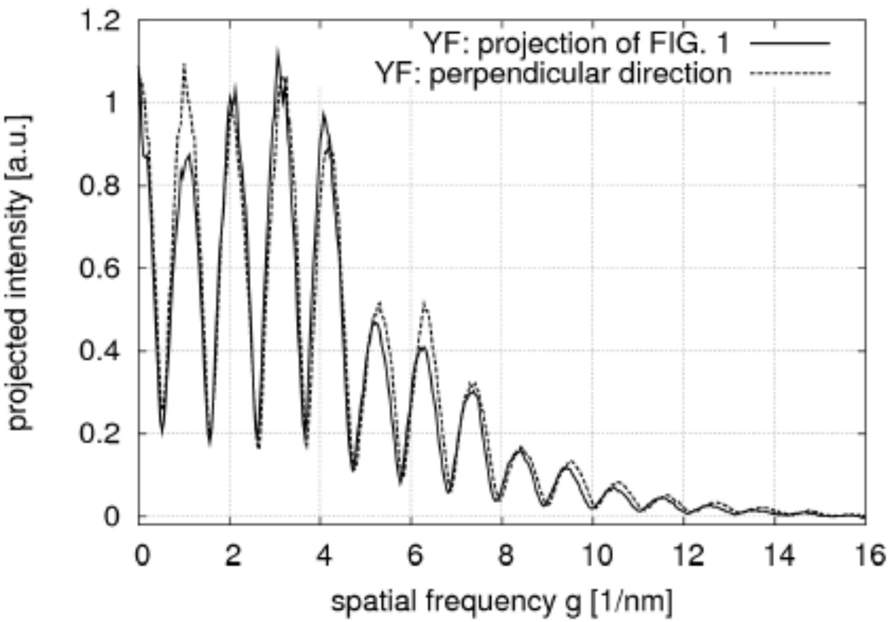


Problem – Dynamic diffraction (coherent information transfer) and the intensity decreases with scattering angle

Young Fringe Measurement of STEHM's Specimen Information Transfer



Young's fringe pattern from tungsten specimen recorded at 300 kV for 4s using STEHM image having Nyquist frequency of 38.3 nm^{-1}.



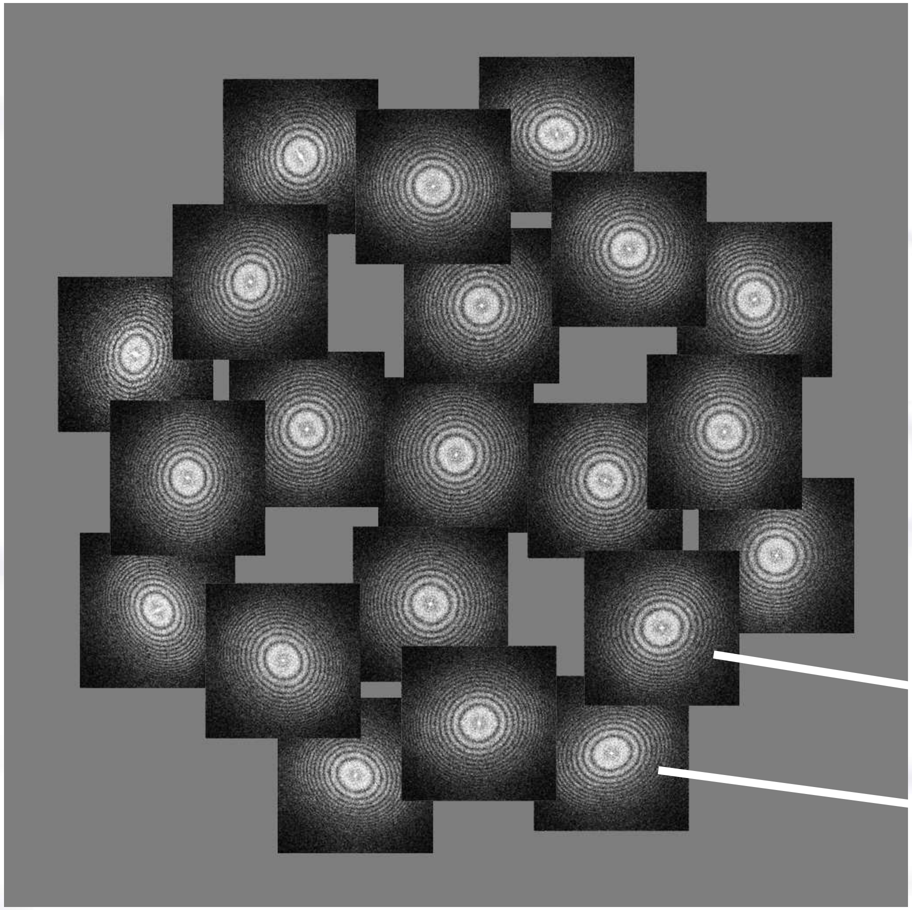
Projected fringe contrast for two perpendicular directions from the Young's fringe pattern.

Problem - Intensity decreases with scattering angle – improved measurement possible using DBI of amorphous materials

H. Müller et al., Nuclear Inst. and Methods in Physics Research A V645 (2011) 20.

STEHM's Aplanatic TEM
(Correction of Spherical and Coma Aberrations)

Necessary for determining coherence properties of quasiparticles
- **phonons**
- **plasmons**
- **magnons**
- **etc.**



Increases HR imaging field of view from 100 nm to 1 mm.

45 mrad

60 mrad

Tilt tableau with an outer tilt angle of 60mrad. The individual positions of the 21 diffractograms corresponds to the position of the illumination tilt.

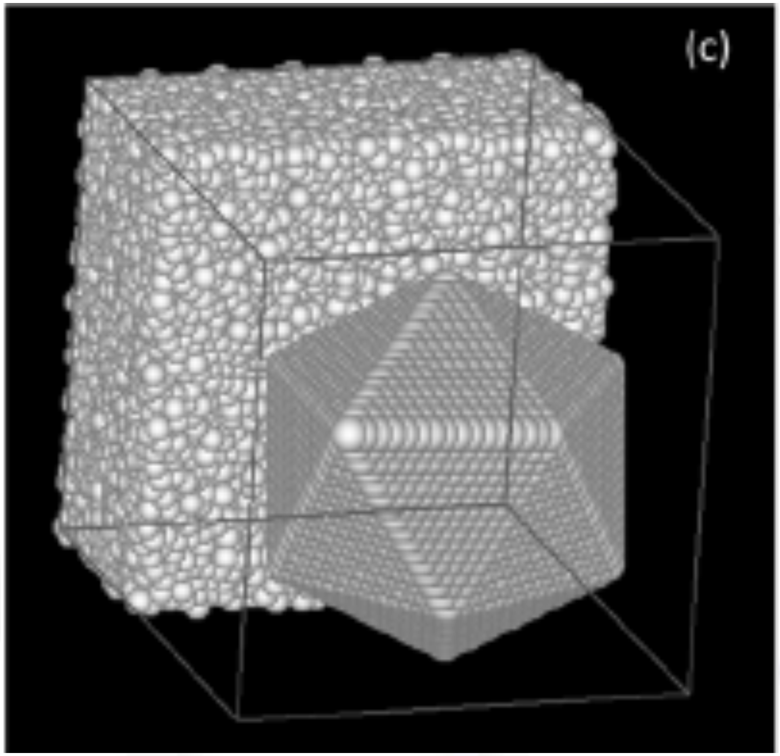
Electron Holography

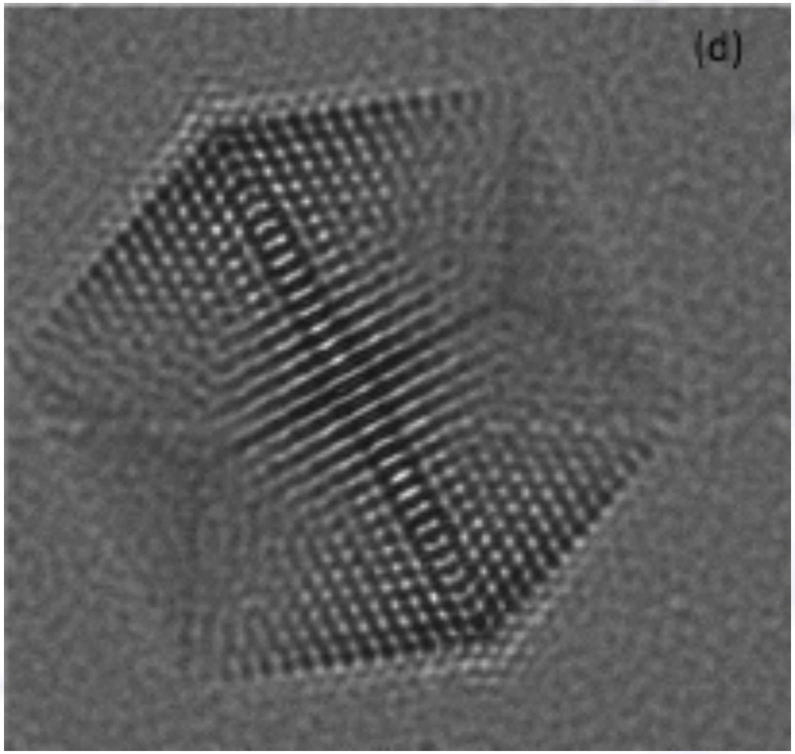
Visualizing the "unseen" world.

Possible to see at the atomic scale:

- magnetic fields
- electrostatic fields
- strain fields
- temperature
- composition
- identify type and number of atoms in lattice image

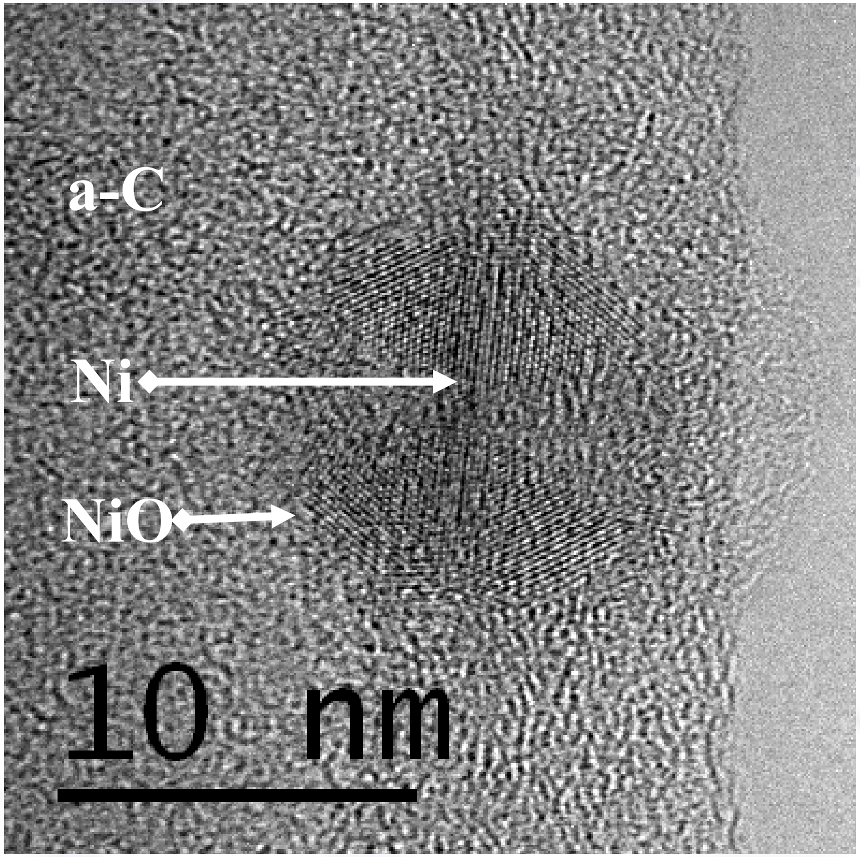
STEHM's Images of Core Shell Ni/NiO Nanoparticles

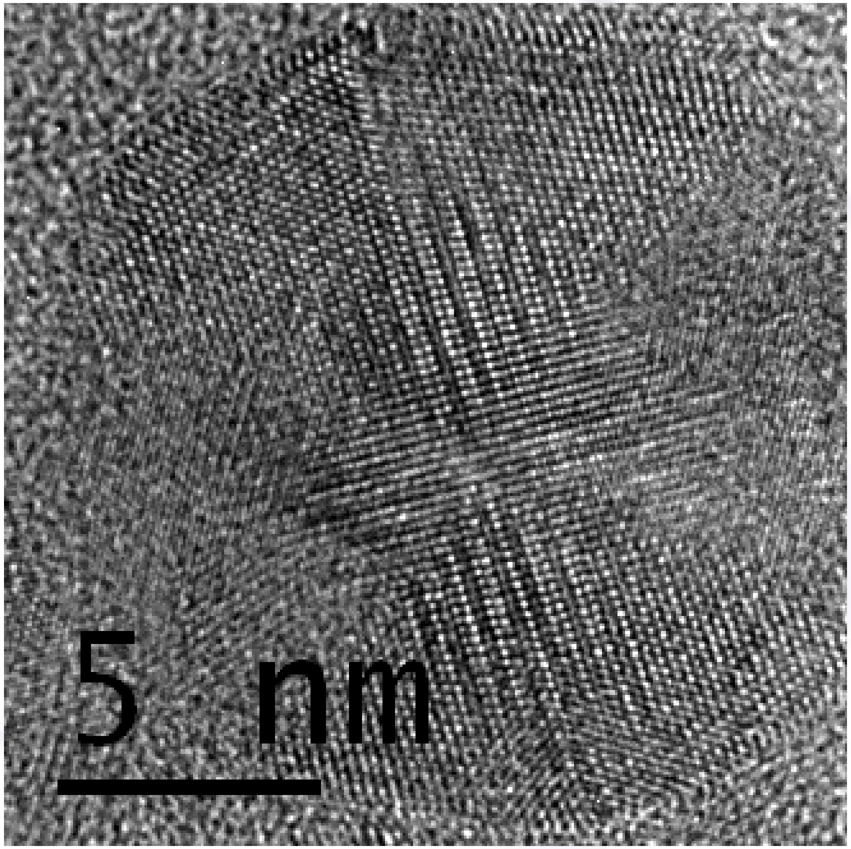




Simulated icosahedral Ni nanoparticle on amorphous Carbon substrate

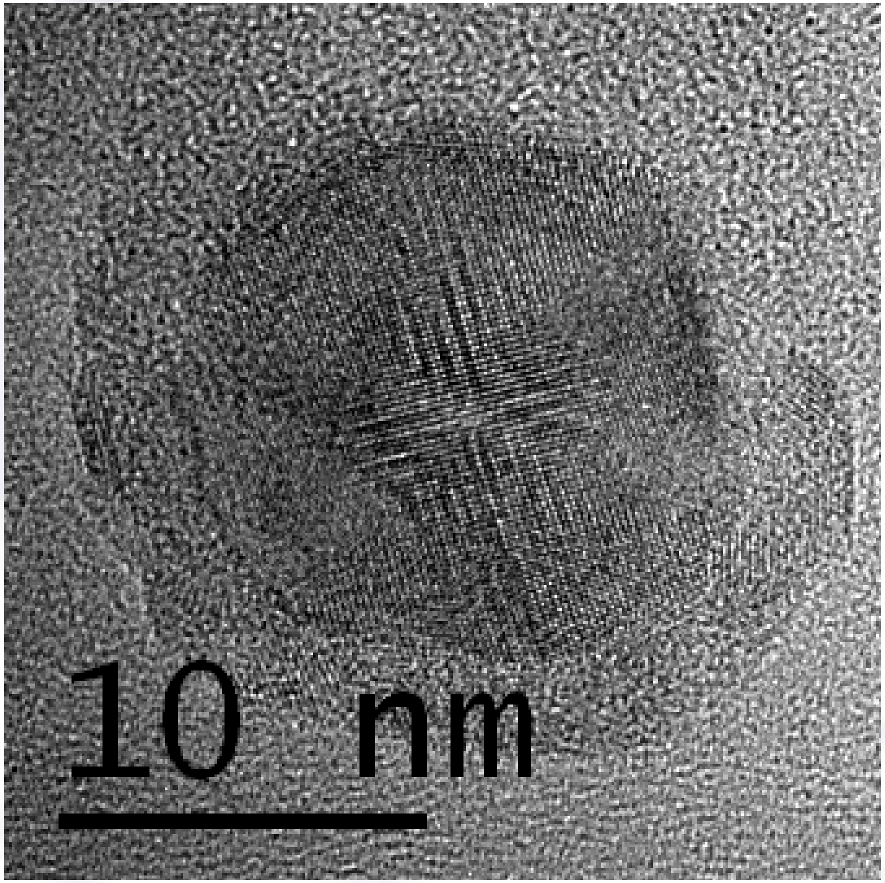
STEHM's Images of Core Shell Ni/NiO Nanoparticles

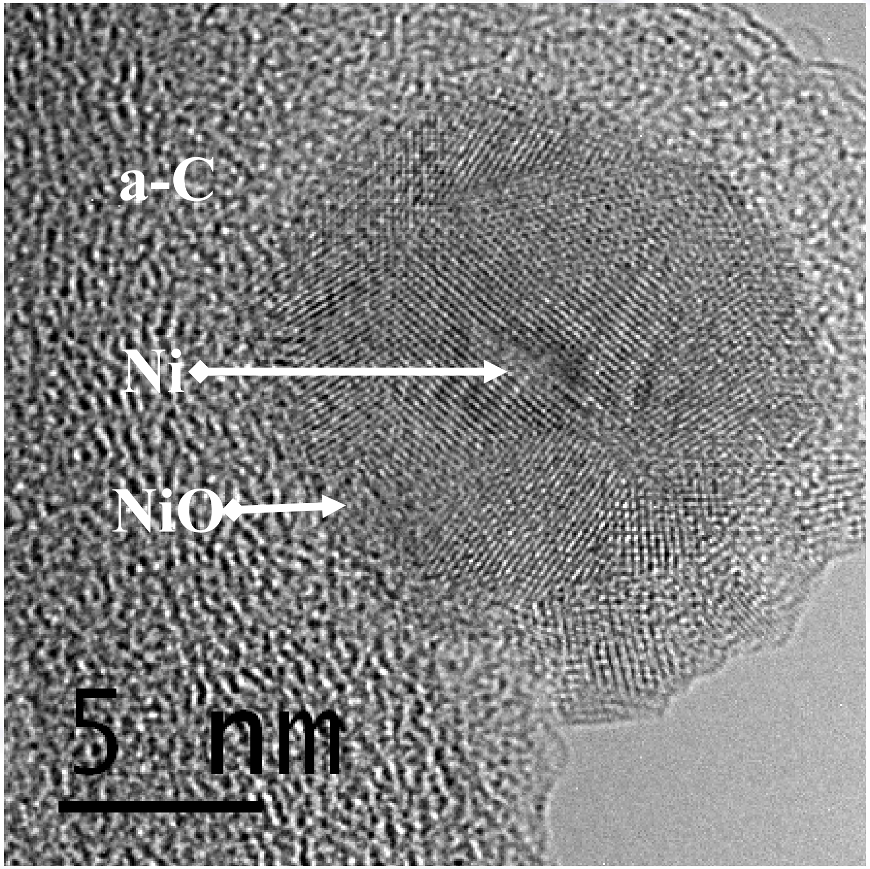




STEHM image of Ni/NiO nanoparticle on amorphous Carbon substrate

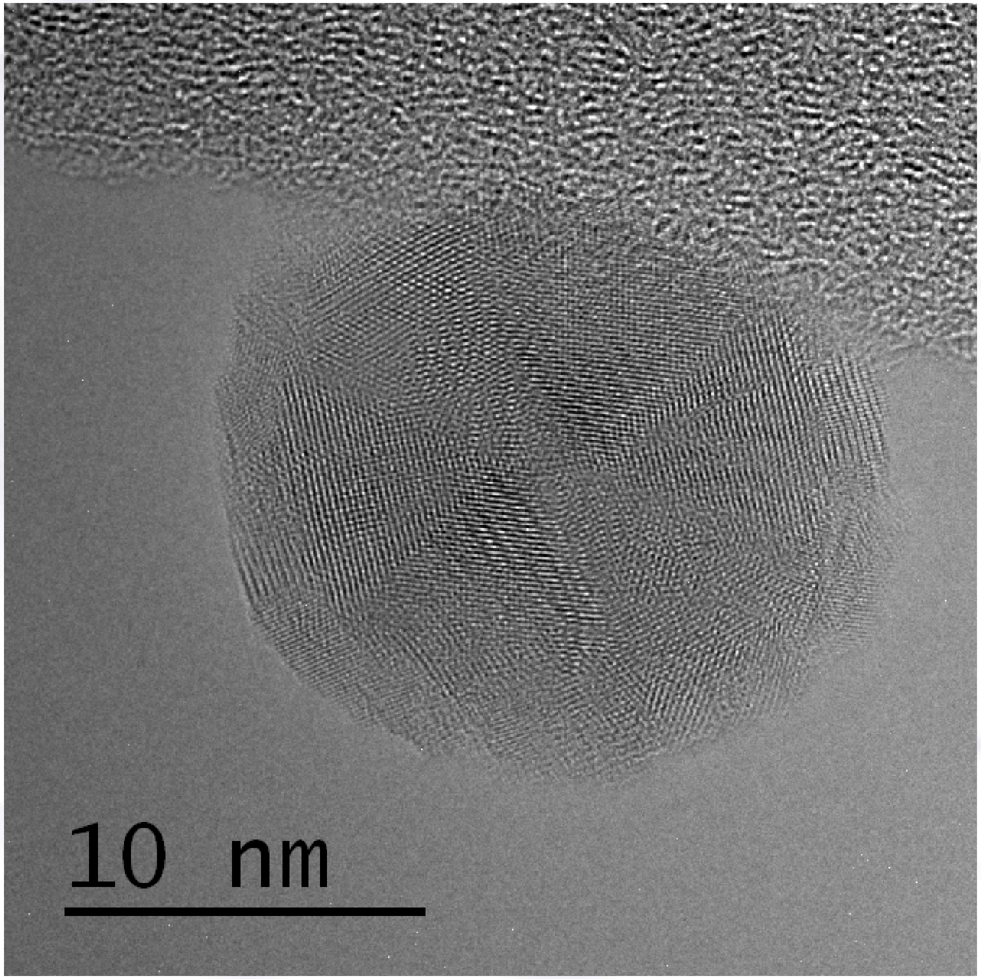
STEHM's Images of Core Shell Ni/NiO Nanoparticles





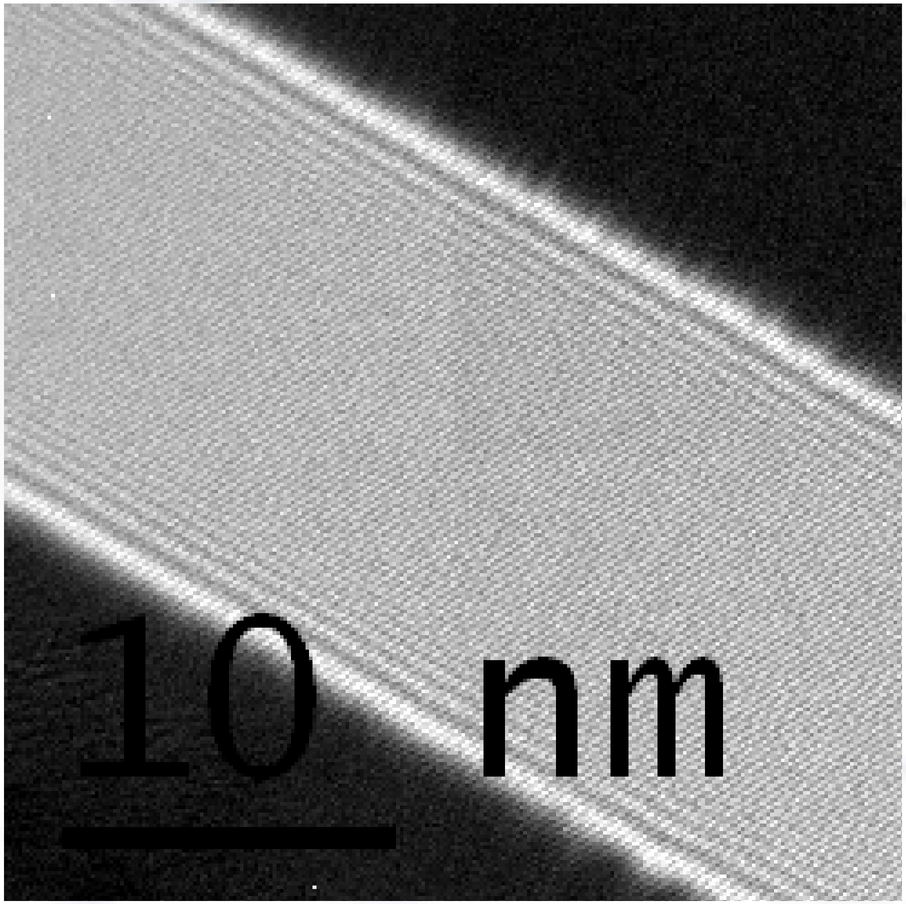
STEHM image of Ni/NiO nanoparticle on amorphous Carbon substrate

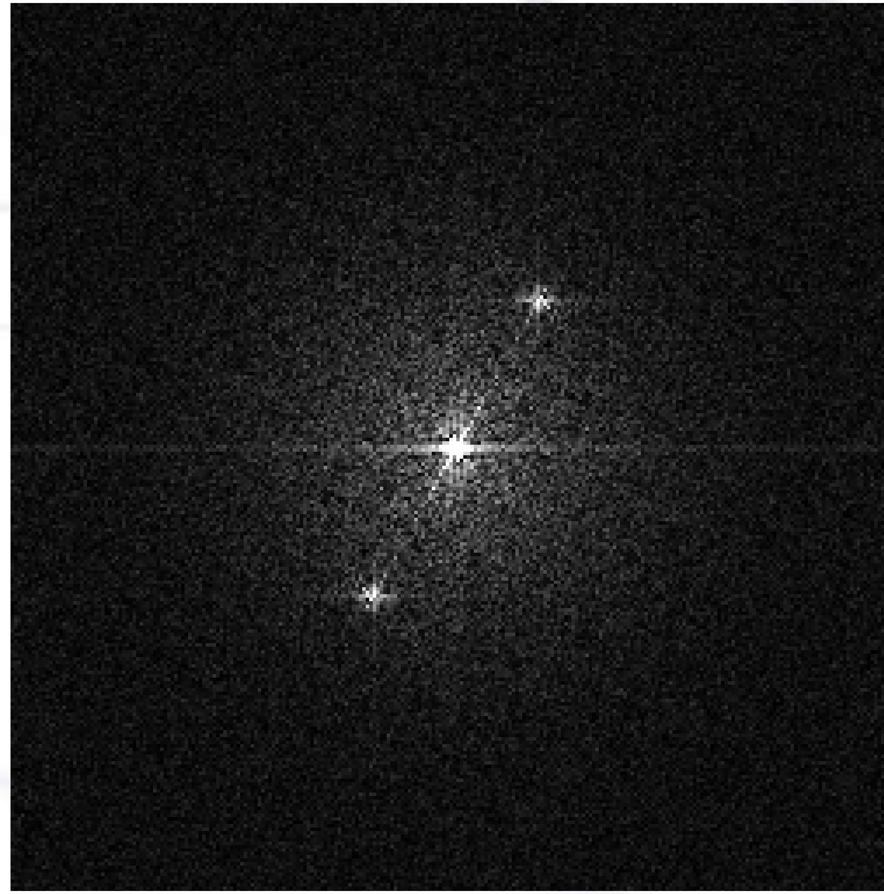
STEHM's Images of Core Shell Ni/NiO Nanoparticles



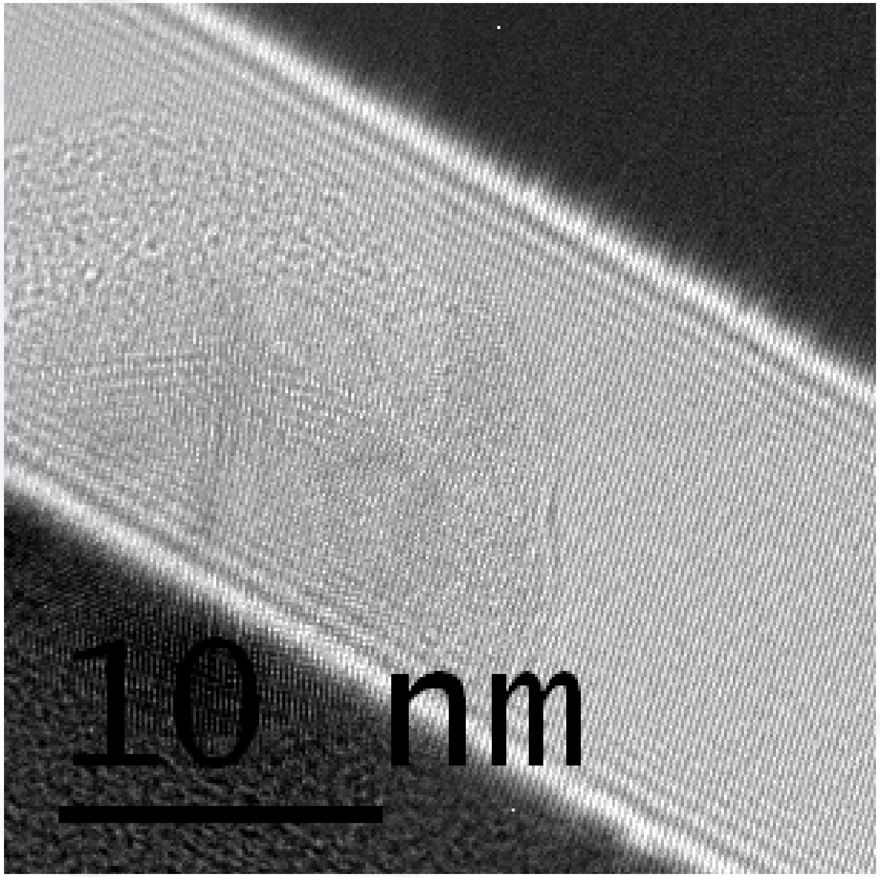
Please use SiNitride substrate to support specimen instead of a-carbon to reduce background noise.

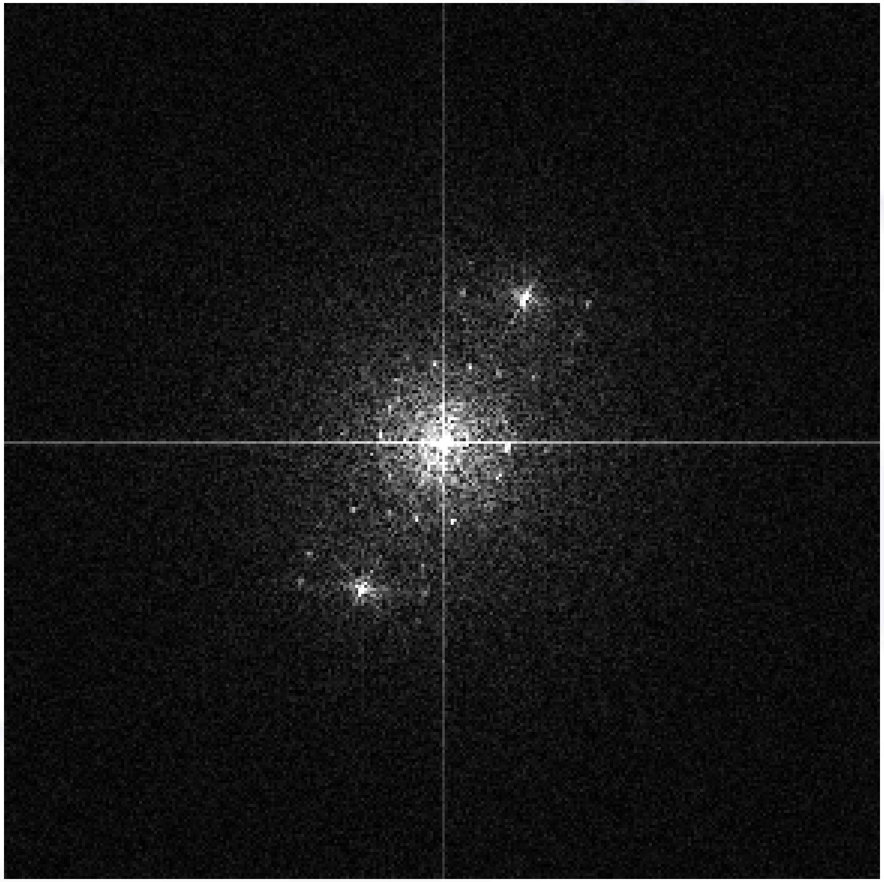
Reference (Empty) Hologram



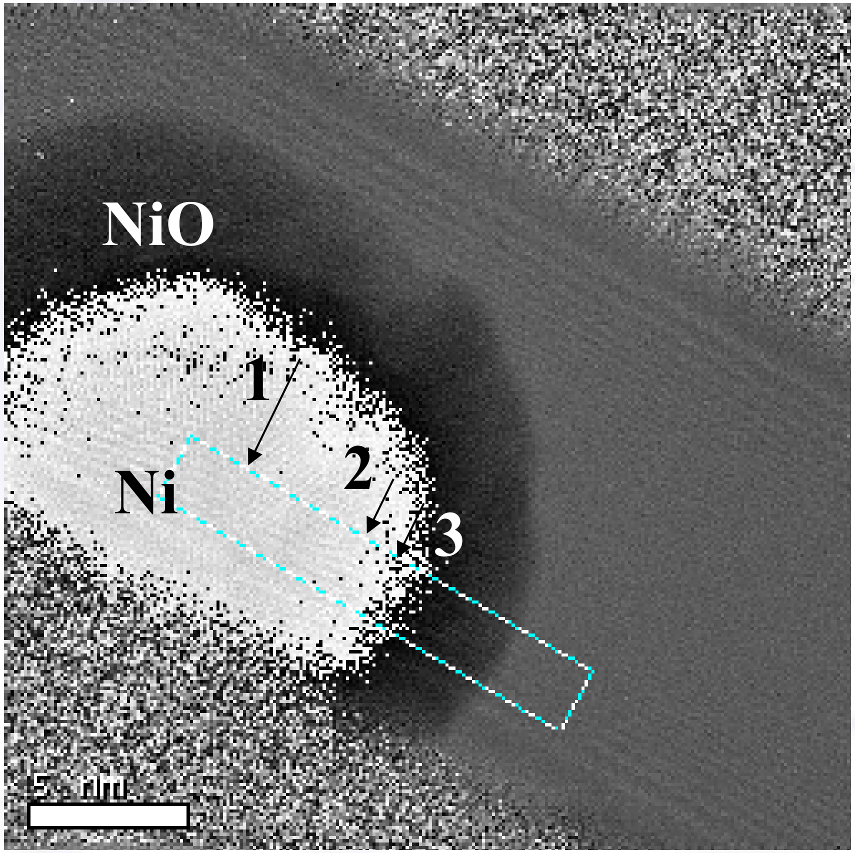


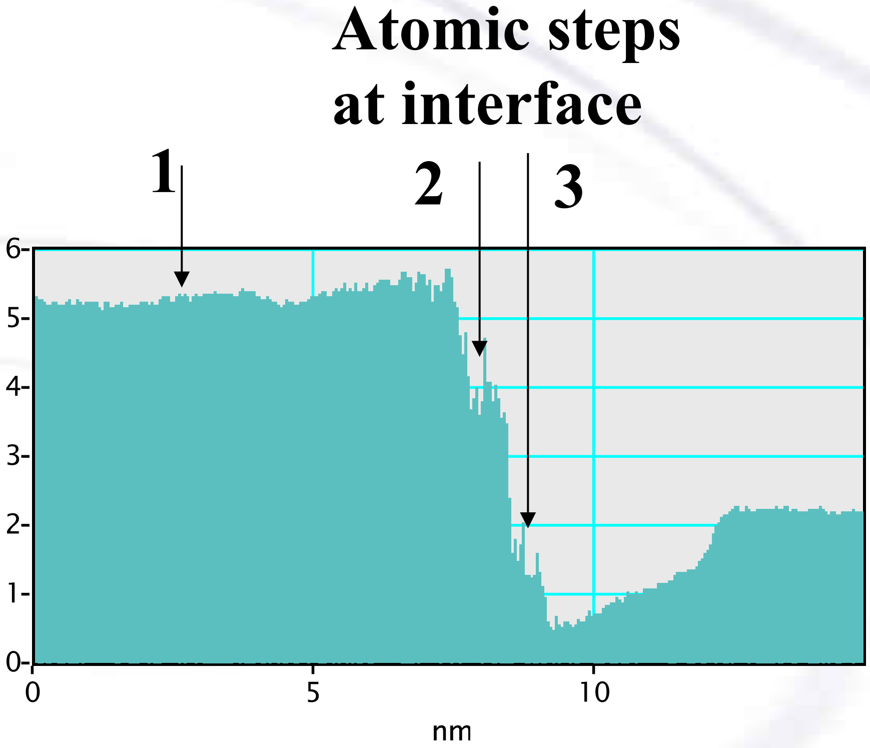
STEHM's Hologram Images of Core Shell Ni/NiO Nanoparticles



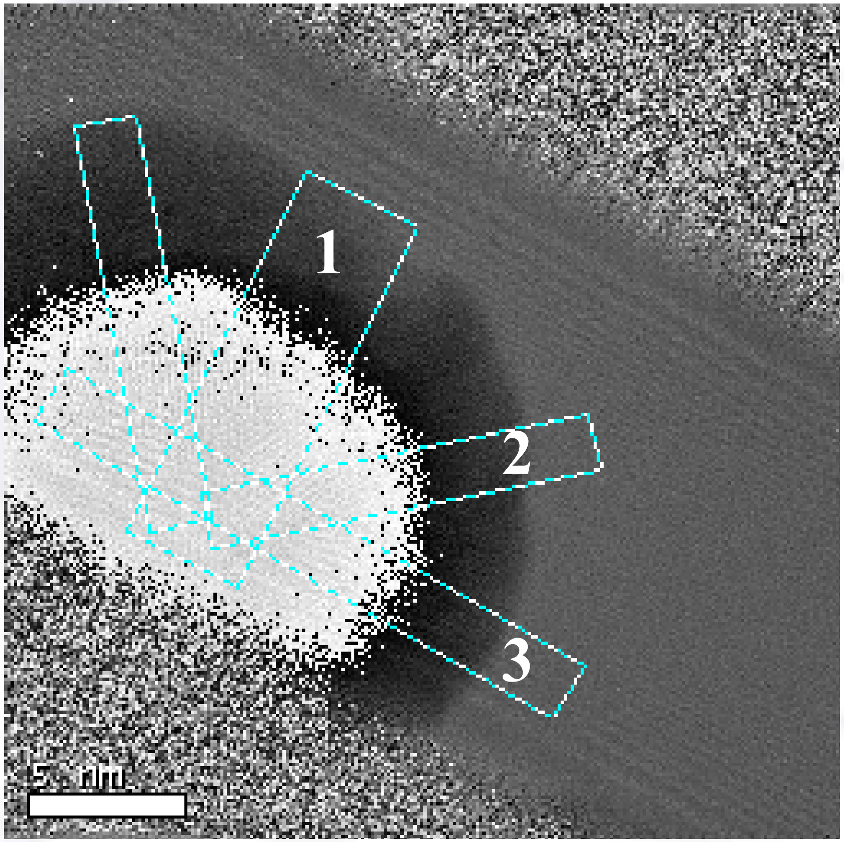


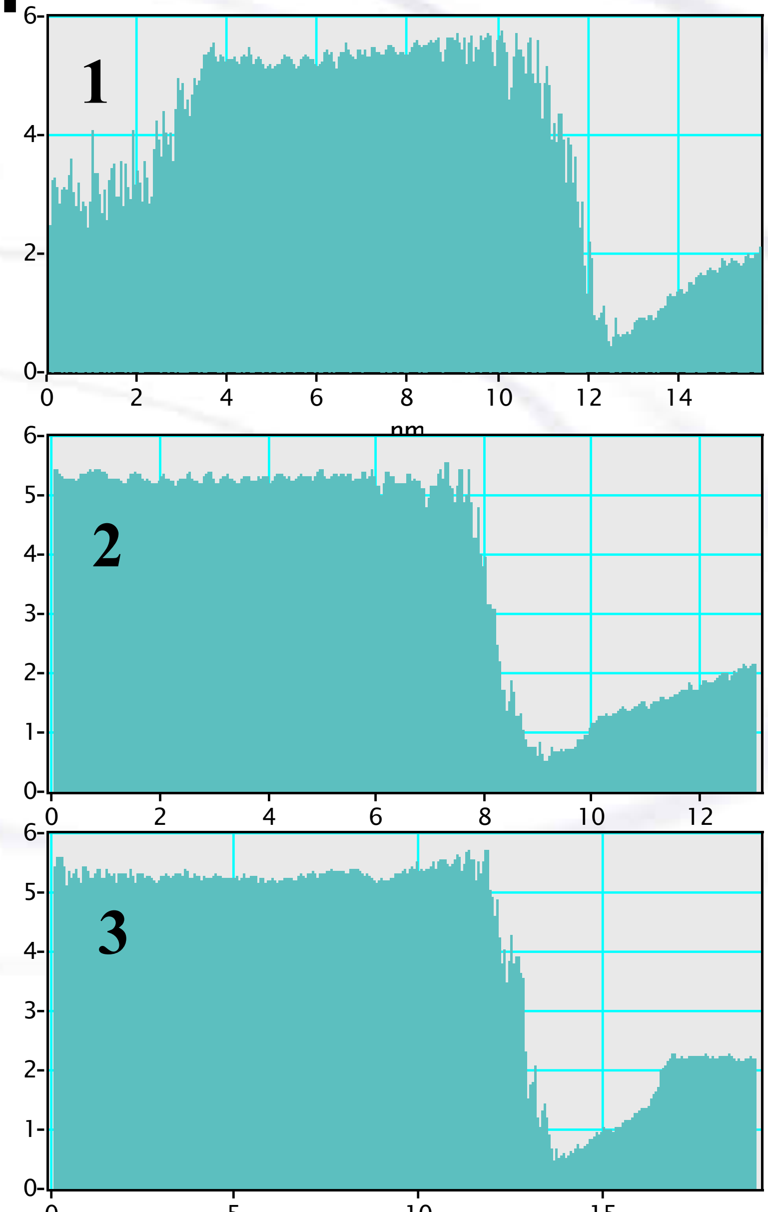
STEHM's Hologram Images of Core Shell Ni/NiO Nanoparticles





STEHM's Hologram Images of Core Shell Ni/NiO Nanoparticles





Ni nanoparticle's morphology appears to have changed due to NiO shell.

Filtered Electron Holography

1) Spatially

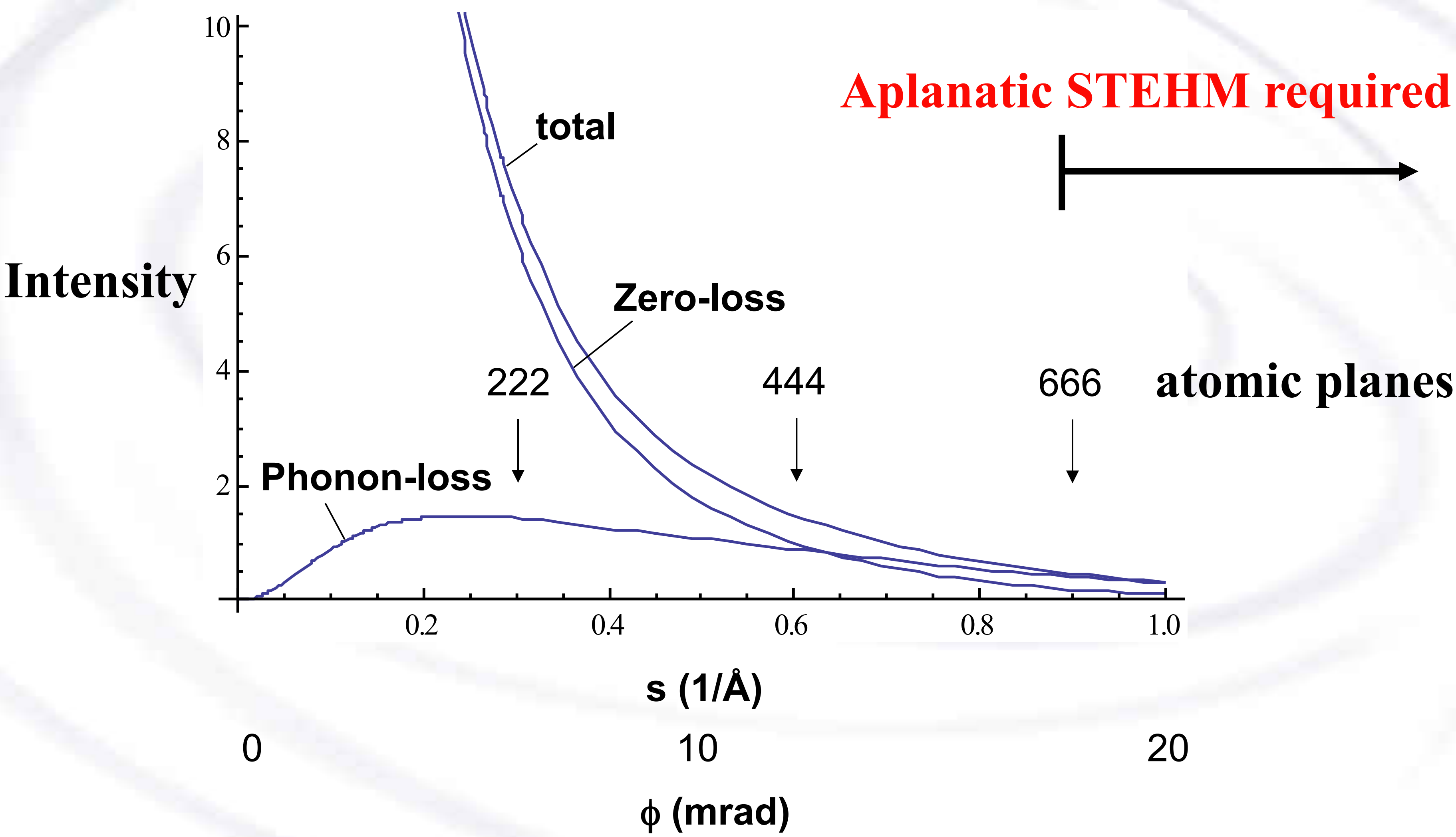
- on diffraction plane

2) Energy-loss

- using energy window
- separate energy loss peak-of-interest from zero- loss peak and other peaks using GIF.

Spatial Filtering on Diffraction Plane

Zero-loss & Phonon-loss Intensities for GaAs



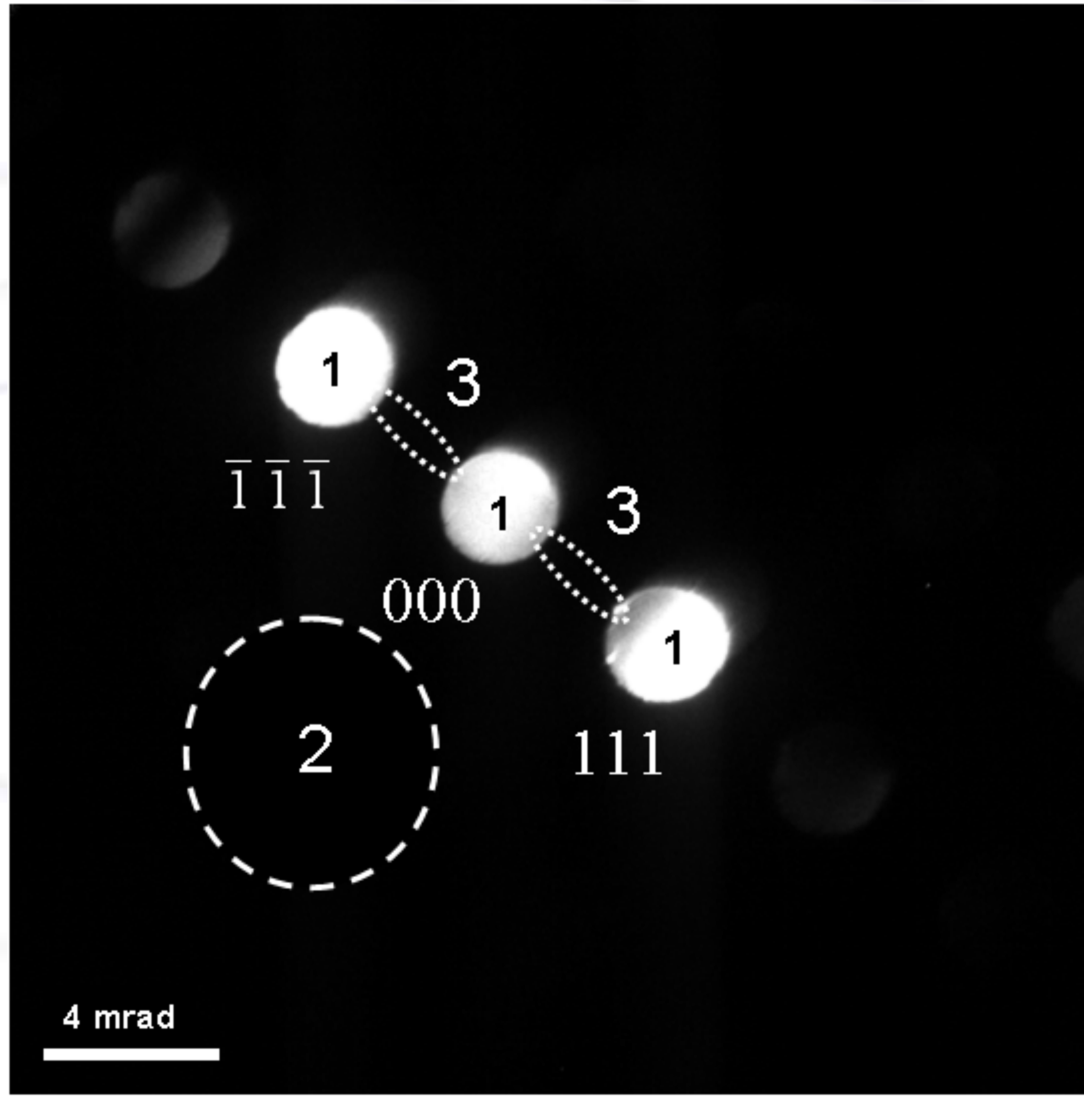
Doyle and Turner Acta Cryst. (1968). A24, 390

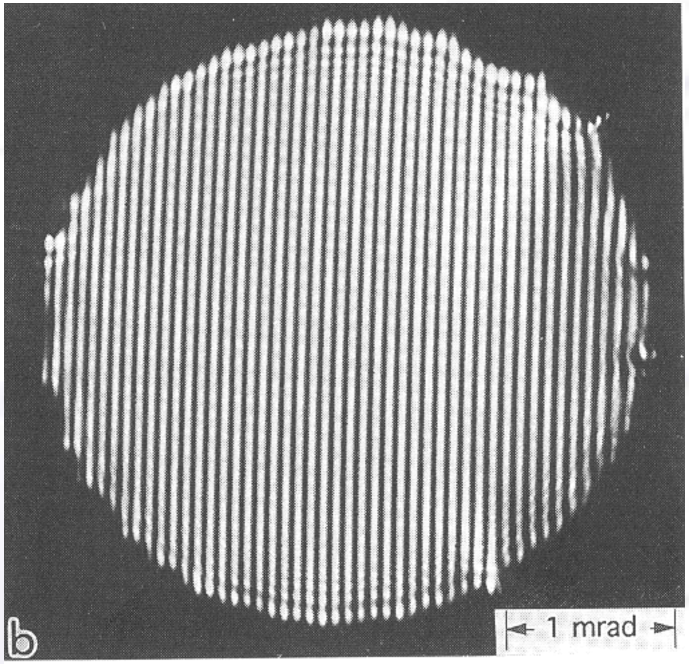
Interfered Regions in Diffraction (k-) Space

1 – elastically scattered, Bragg diffracted beams

2 – inelastically scattered, phonon loss electrons from nuclear core (Z – contrast imaging)

3 – inelastically scattered, phonon loss electrons from thermal vibration of atoms



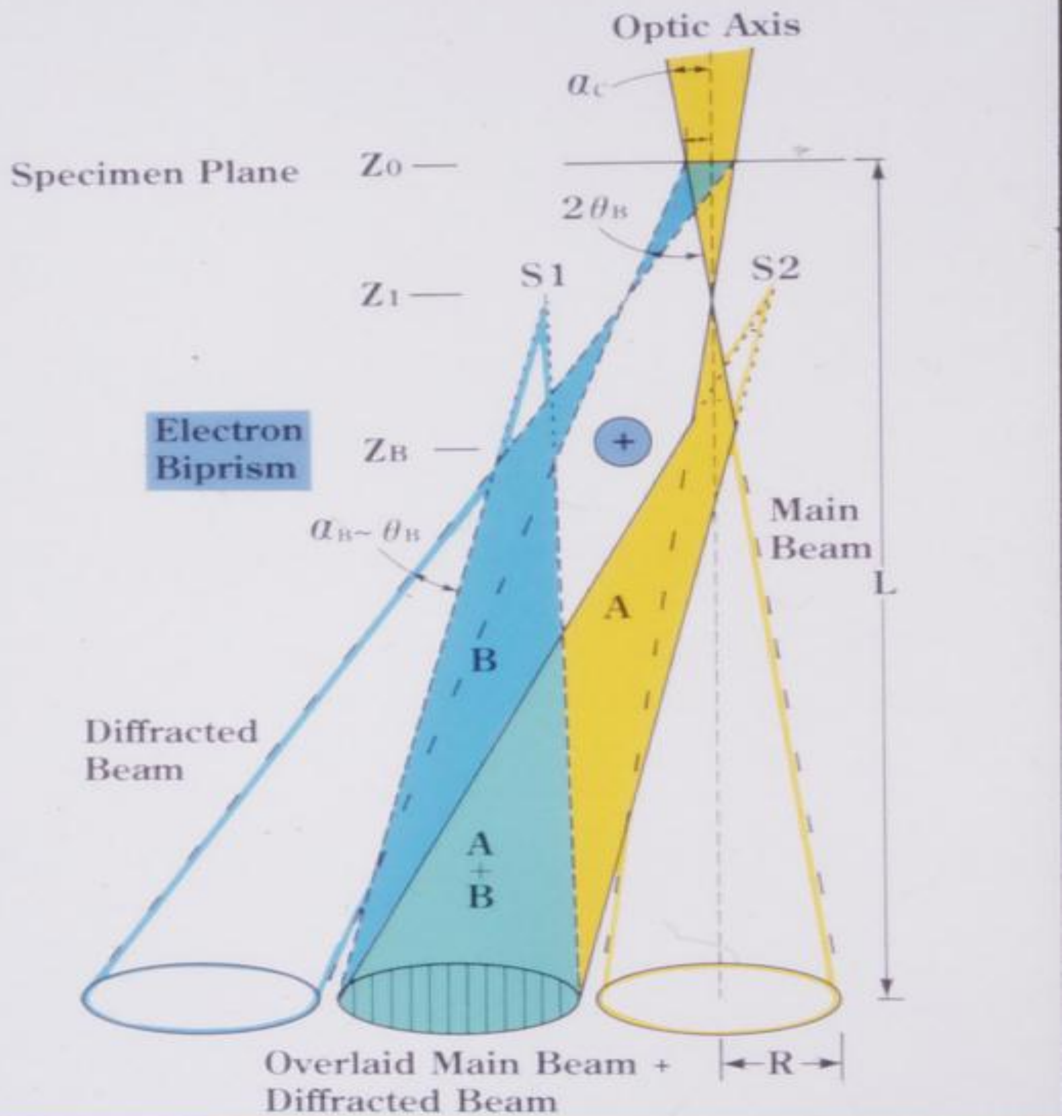


Diffracted Beam Interferometry/ Holography (DBI/H)

Hologram of the main beam (000) interfering with the 111 diffracted beam of GaAs.

Invented during Tonomura Electron Wavefront Project.

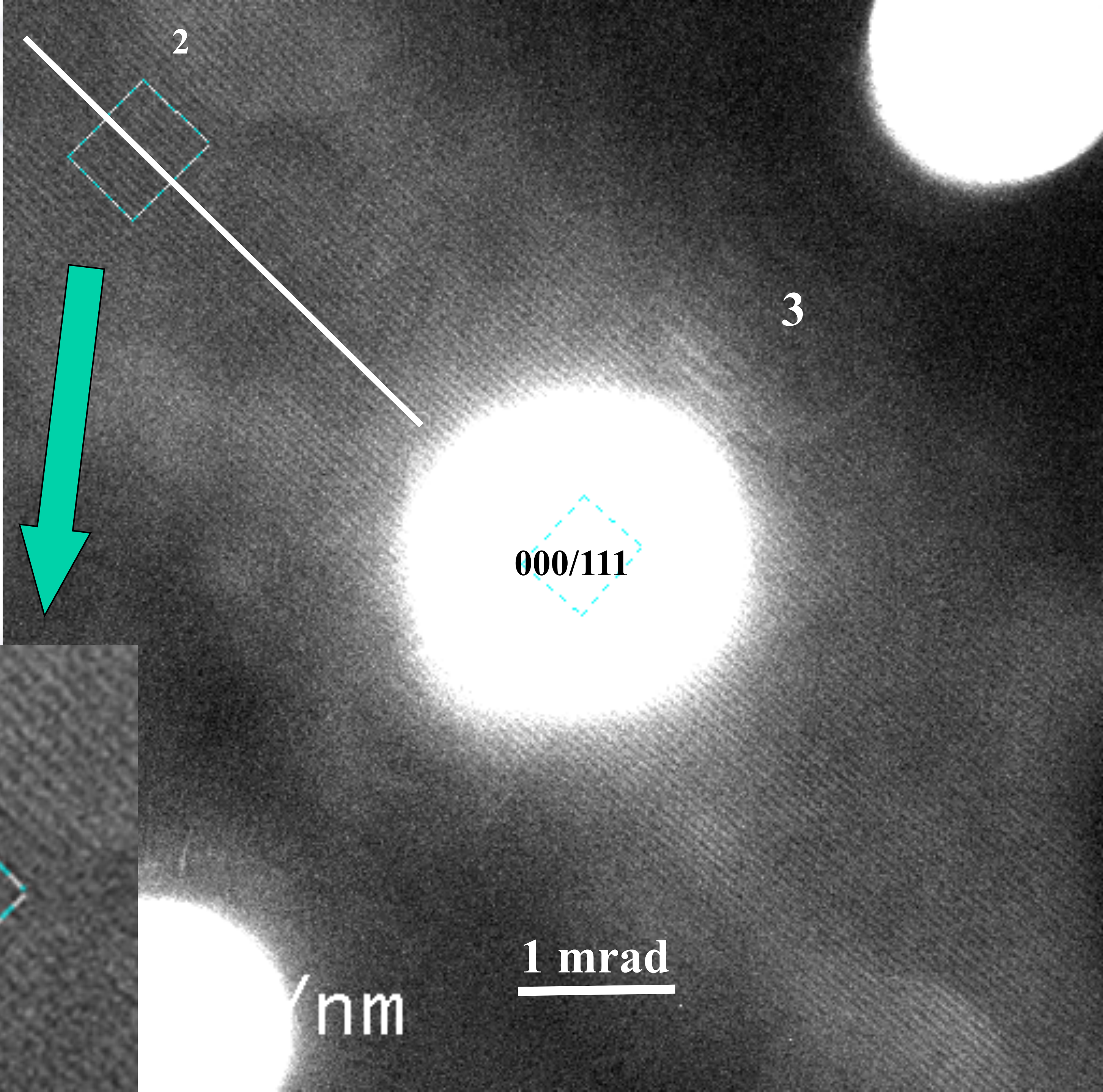
R. A. Herring, G. Pozzi, T. Tanji and A. Tonomura, "Interferometry using convergent beam electron diffracted beams plus an electron biprism" Ultramicroscopy 60 (1995) 153 - 168.

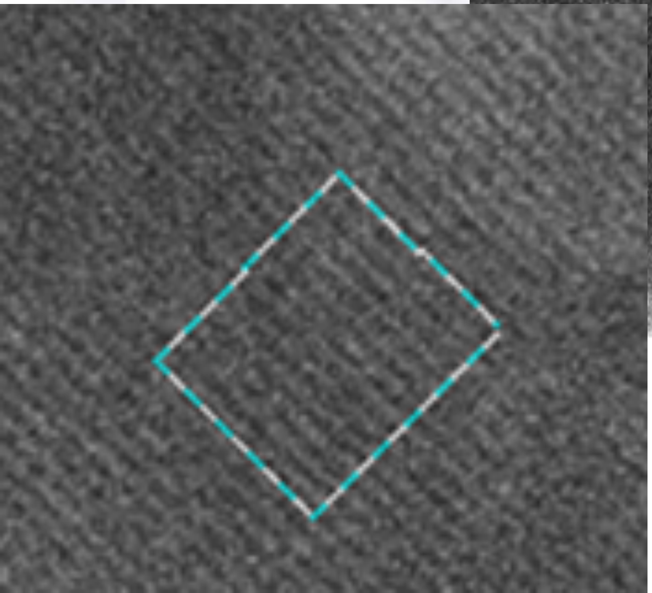


Interference Of Convergent Electron Beams By Use Of An Electron Biprism

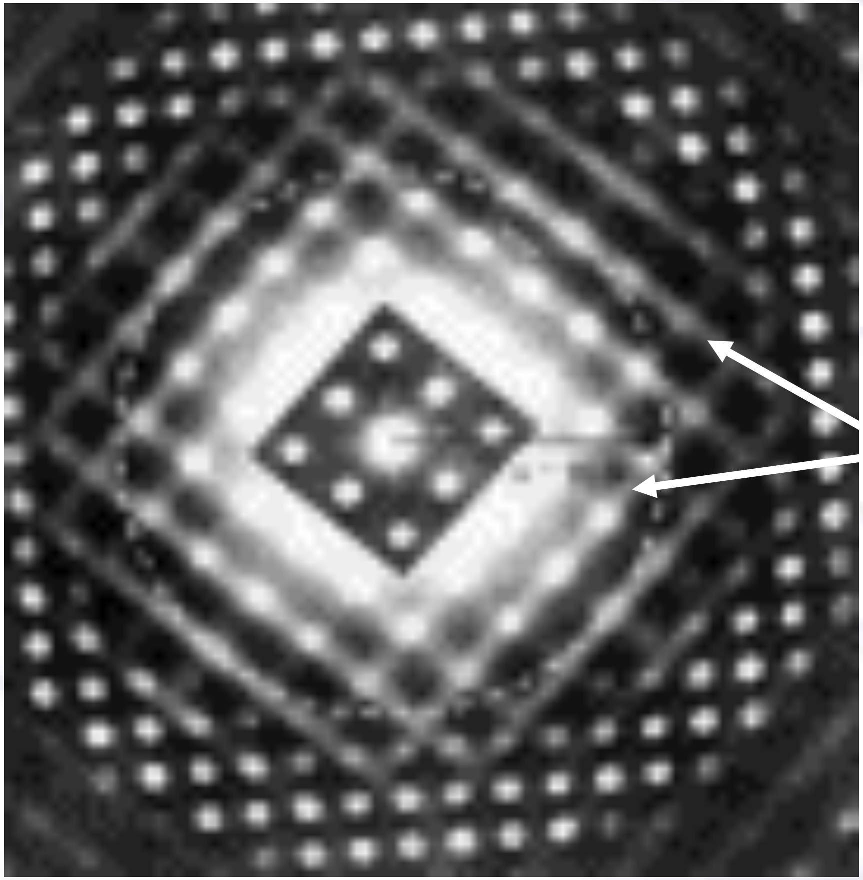
Example

Spatial-filtering of phonon-loss + zero loss electrons generated from a Germanium specimen.





Thermal Diffuse Scattering of Electrons
(low-angle diffusely scattered electrons)



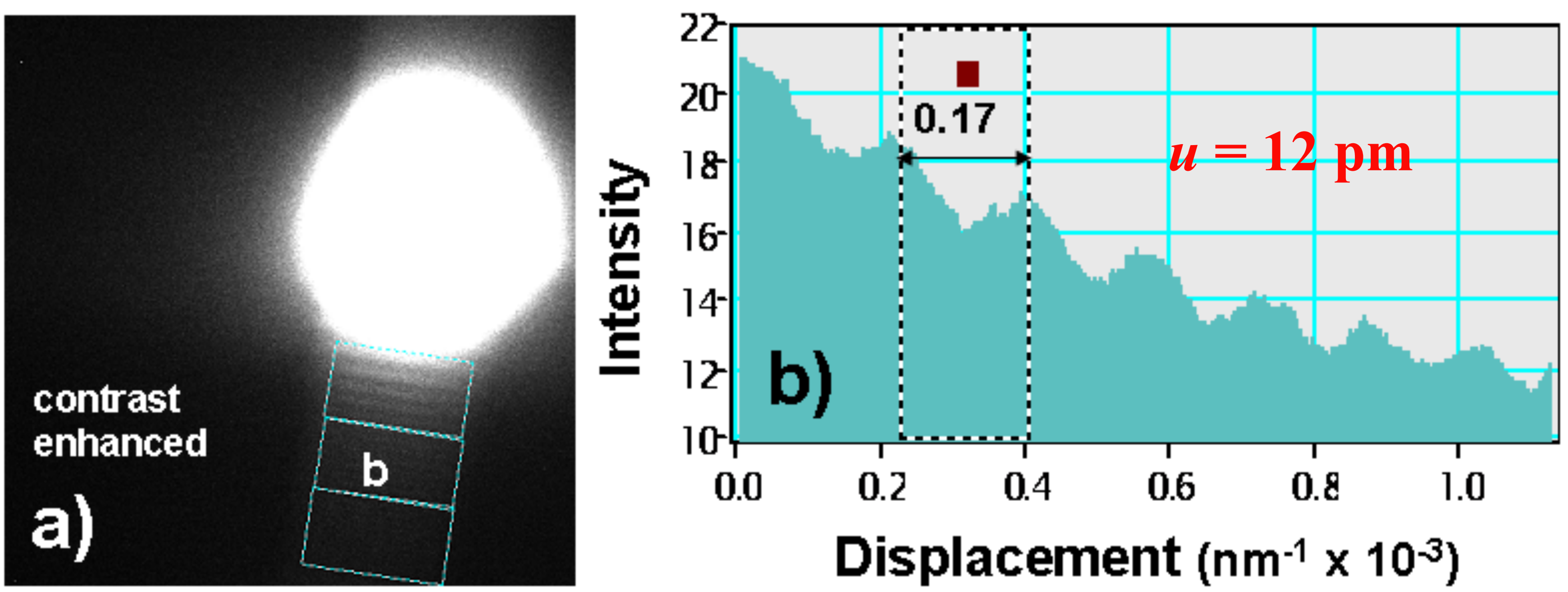
TDS Intensity due to thermal vibration of atoms.

Electron diffraction pattern recorded from GaAs, showing the presence of diffuse scattering streaks between the Bragg reflections due to thermal vibration of the atoms in the crystals.

Z.L. Wang, Micron 34 (2003) 141.

Measurement of Mean Atomic Vibration Amplitude, *u*

$$k(r_1 - r_2) = -16\pi^2 u^2 \sin^2 \theta_B / \lambda^2$$



a) Interference of the 000 and 222 beams of Aluminum resulting in the formation of fringes in the intensity of region b shown in b) used to measure the mean displacement of atoms, *u*, of 12 pm - a first time measurement.

Herring, Microscopy 62 (2013) S99 – S108.

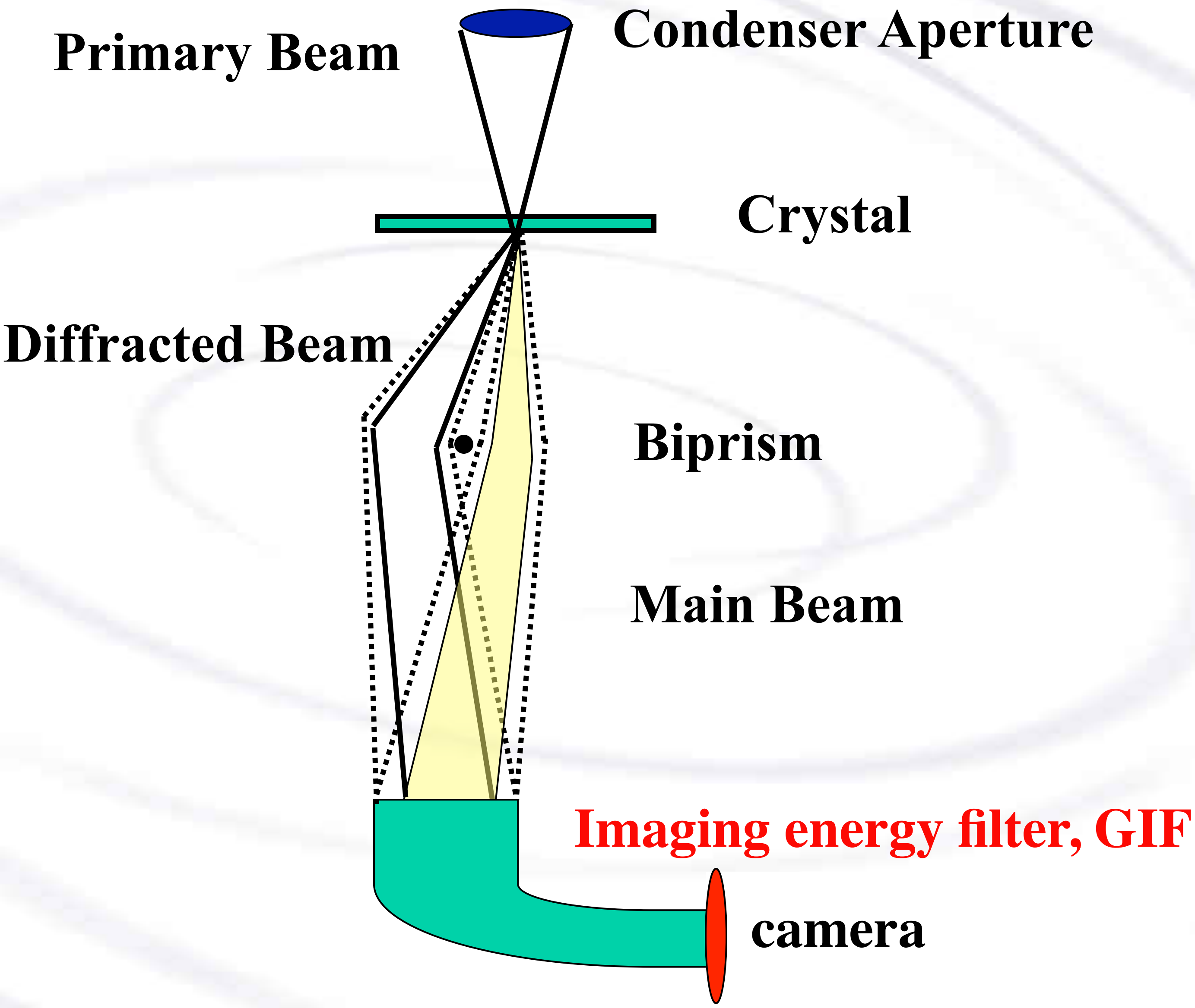
Mean Atomic Vibration Amplitude, *u*

Can be used to:

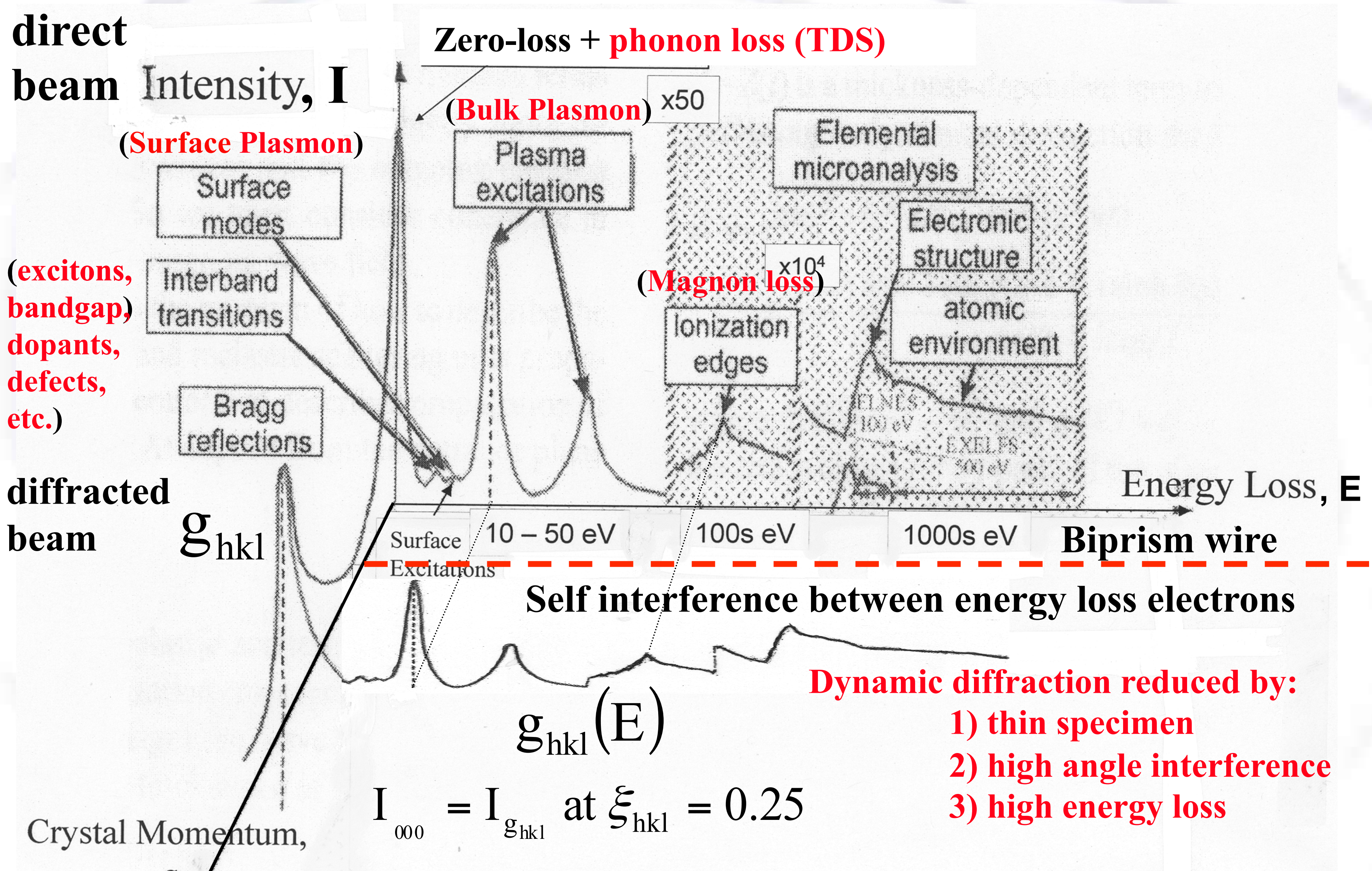
- determine energy of molecular dynamic reactions

- measurement of time using frequency of atomic vibrations

Energy-Filtered DBI/H



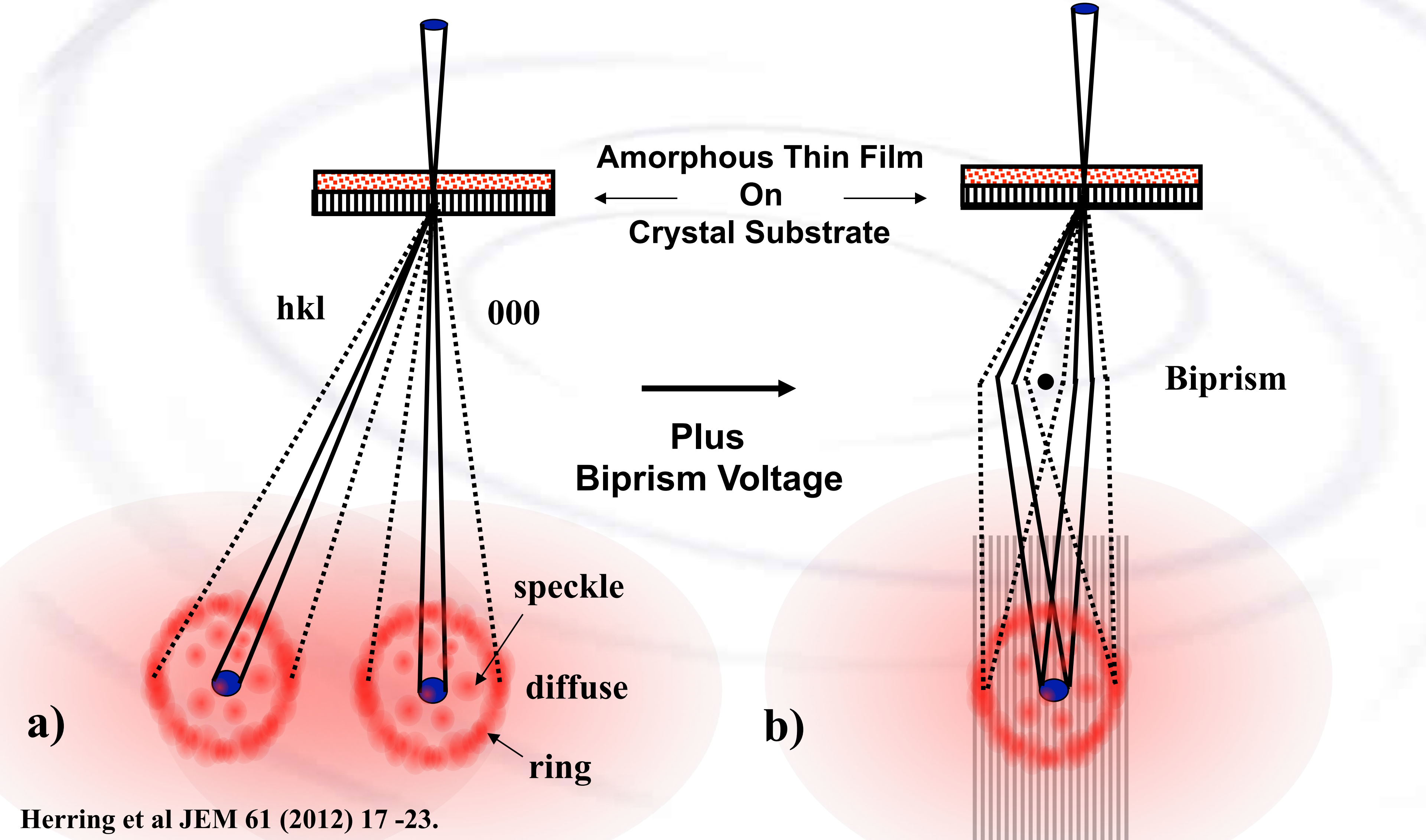
Self-Interference of Elastically & Inelastically Scattered Electrons



Modified from Schattschneider and Werner (2005) *J. Electron. Spectrosc. Relat. Phenom.* 143: 81

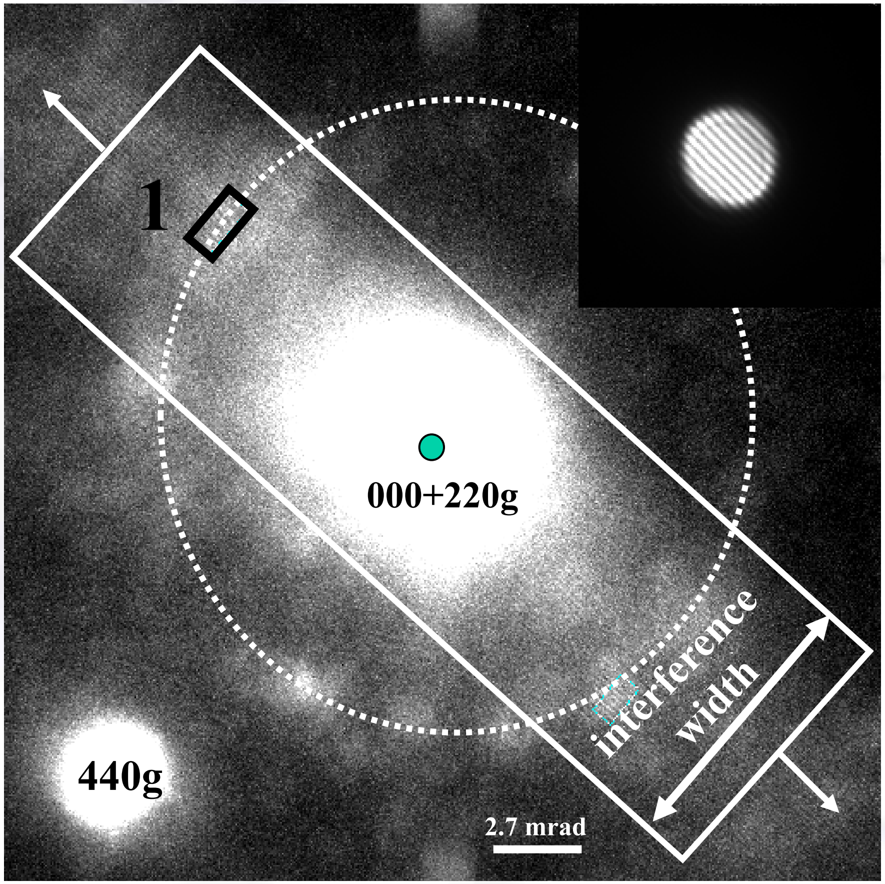
Self-Interference of Amorphous Surface Layer Intensity

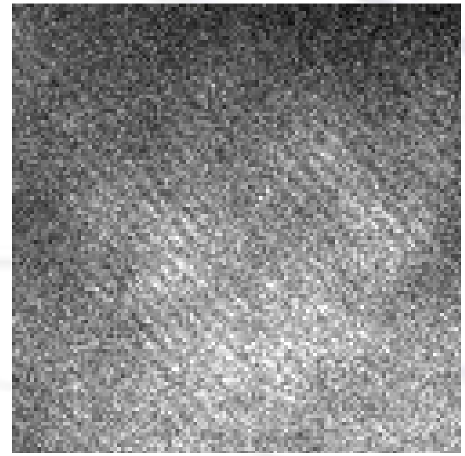
$$\phi = \beta_{g_{hkl}} + \beta_{amorph} - \beta_{g_{-h-k-l}} + \beta_{amorph} = 2\beta_{amorph}$$



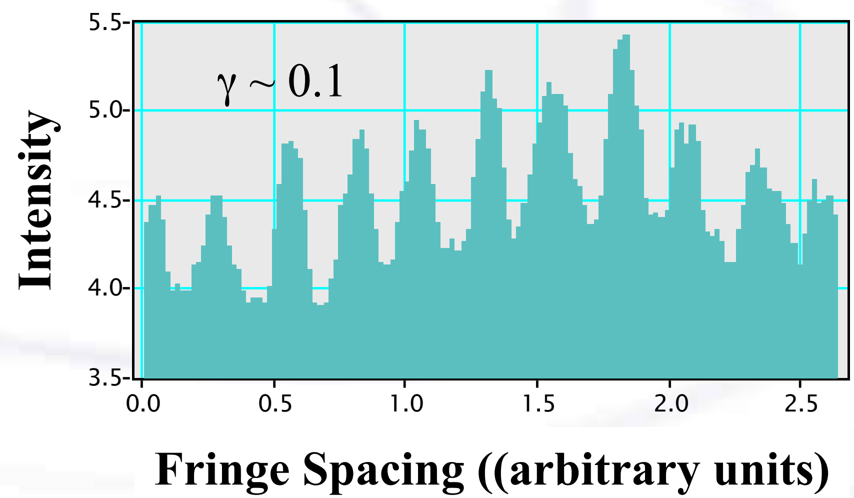
Herring et al JEM 61 (2012) 17 -23.

Phase Measurement of Amorphous GaAs Material





Fringes at 1



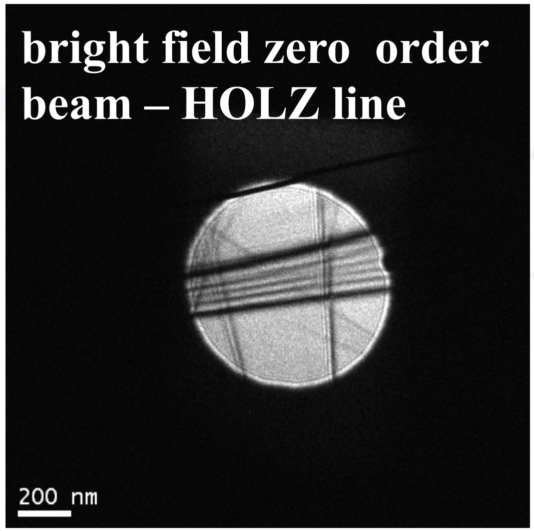
Fringe Contrast at 1

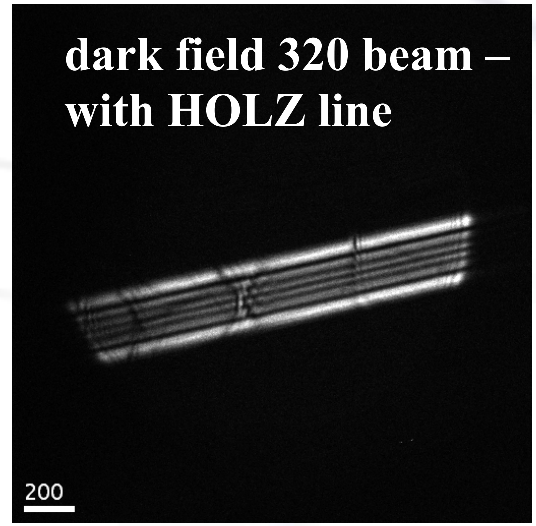
$$\phi = 2\beta_{amorph}$$

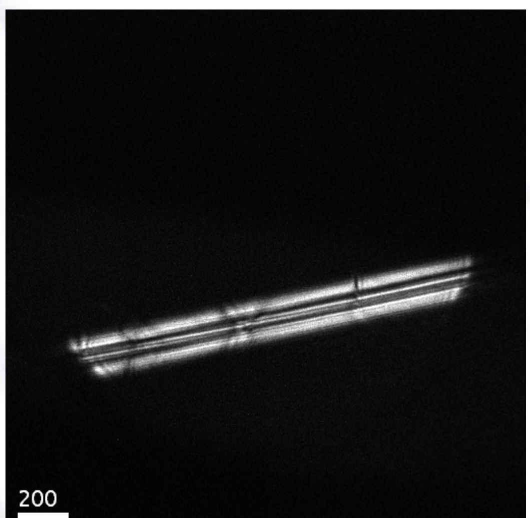
Herring, Saitoh, Tanji, Tanaka JEM 61 (2012) 17 -23.

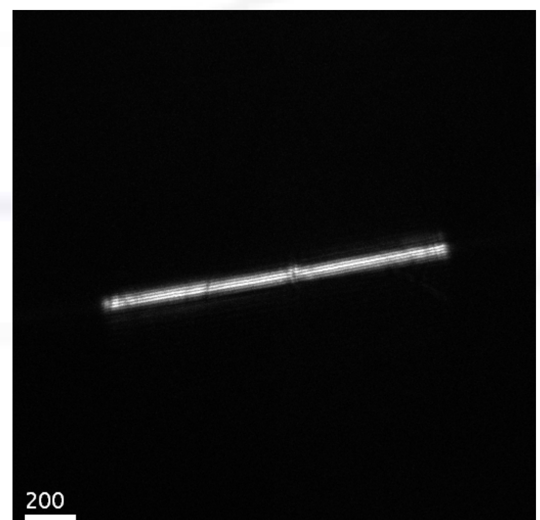
Higher Order Laue Zone (HOLZ) Lines for Internal Strain Measurements

Super-high resolution strain measurements



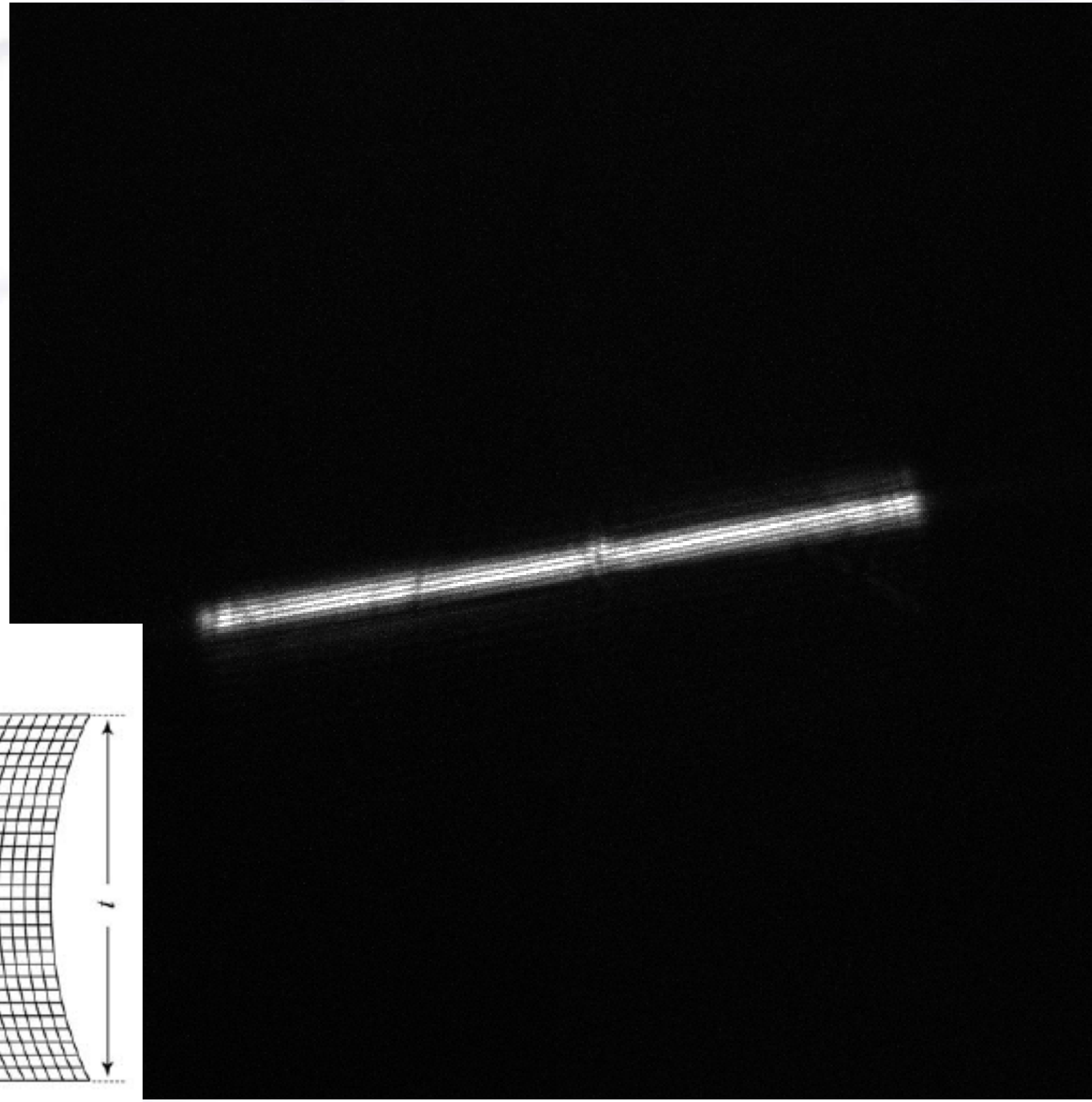


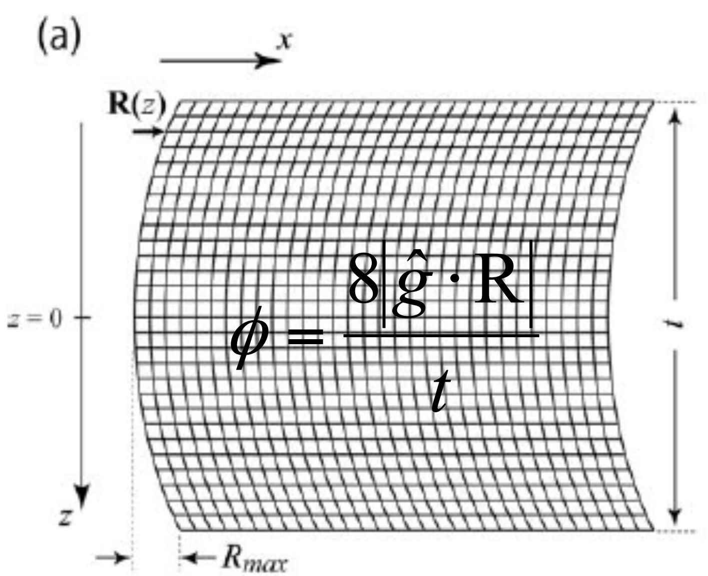




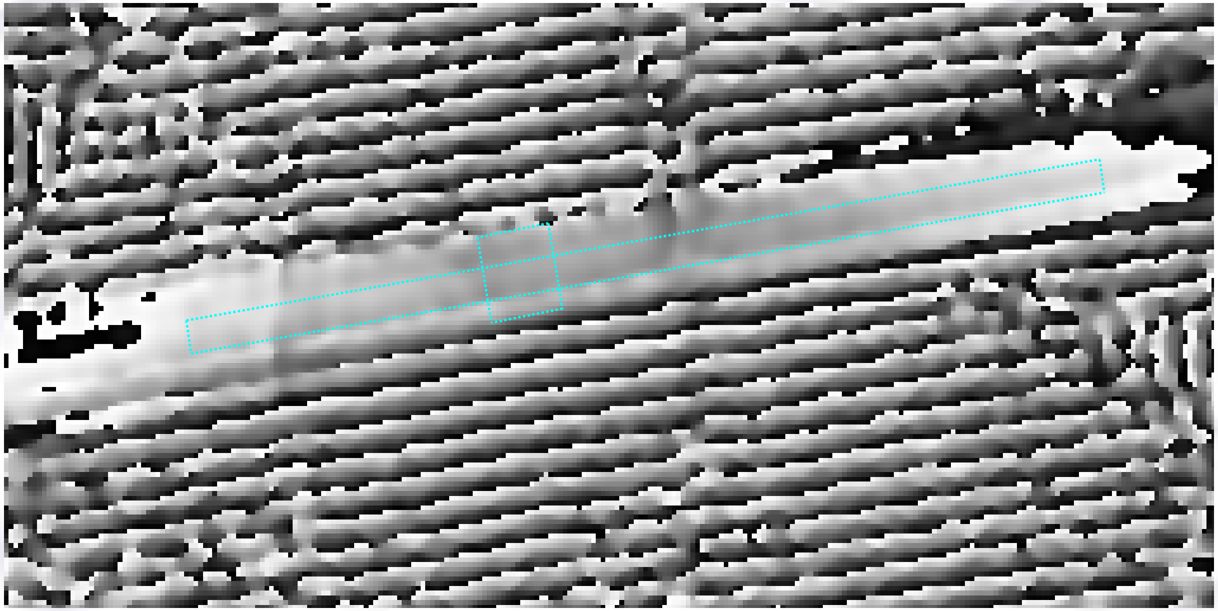
Herring et al Microsc & Microanalysis (Nashville, 2011)

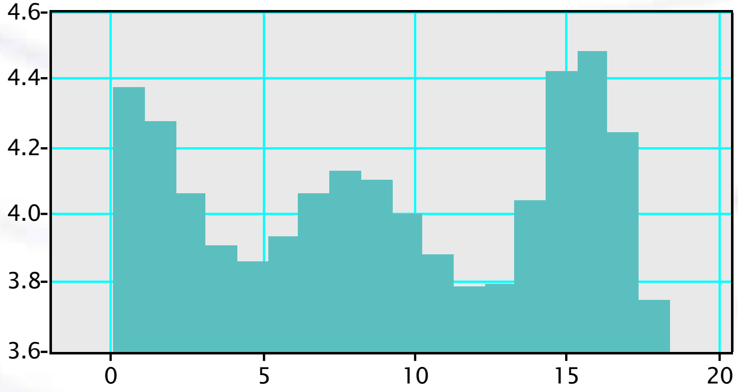
Combined with confocal electron holography for **3D measurements** **of strain**





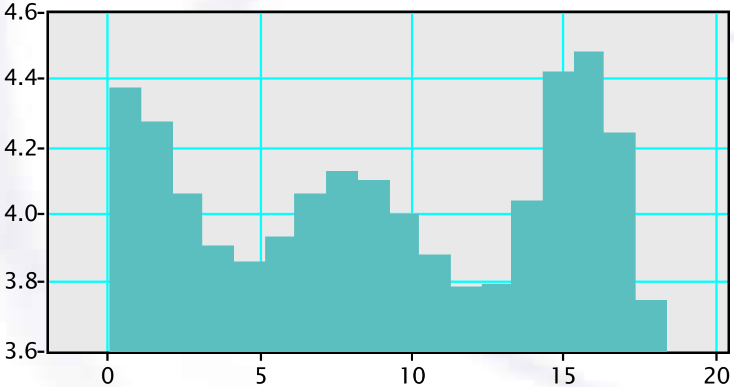
HOLZ Line Phase Measurement



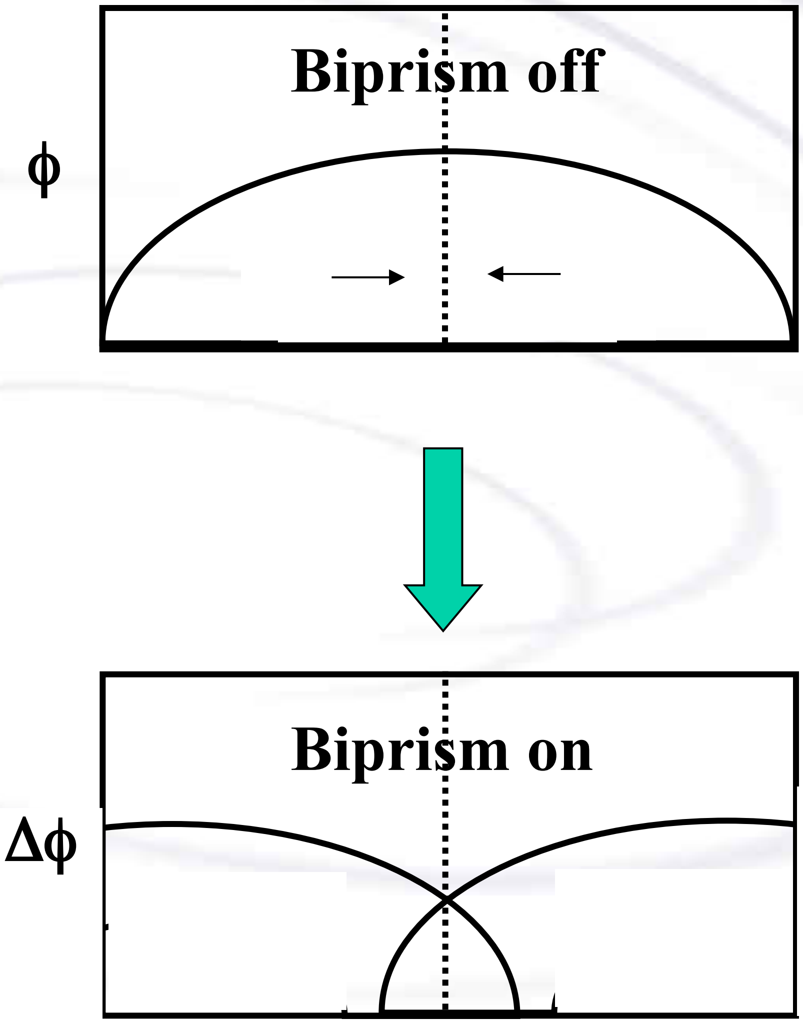


HOLZ Line Phase Measurements

Enables higher-resolution measurement of strain

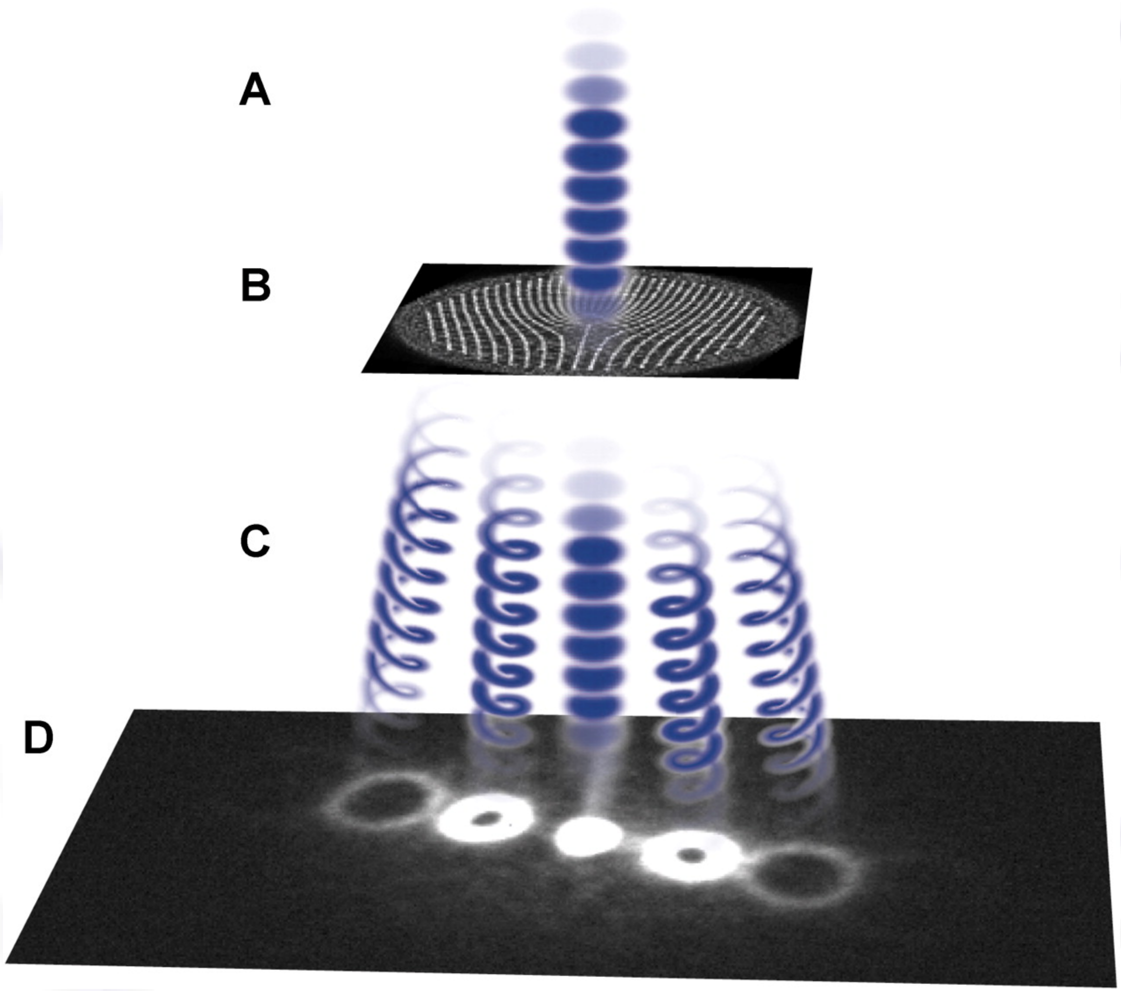


$$\phi = \frac{8\left|\hat{g}\cdot \mathrm{R}\right|}{t}$$



Herring, Saitoh, Tanji, Tanaka, unpublished.

Formation of Electron Vortice Beams



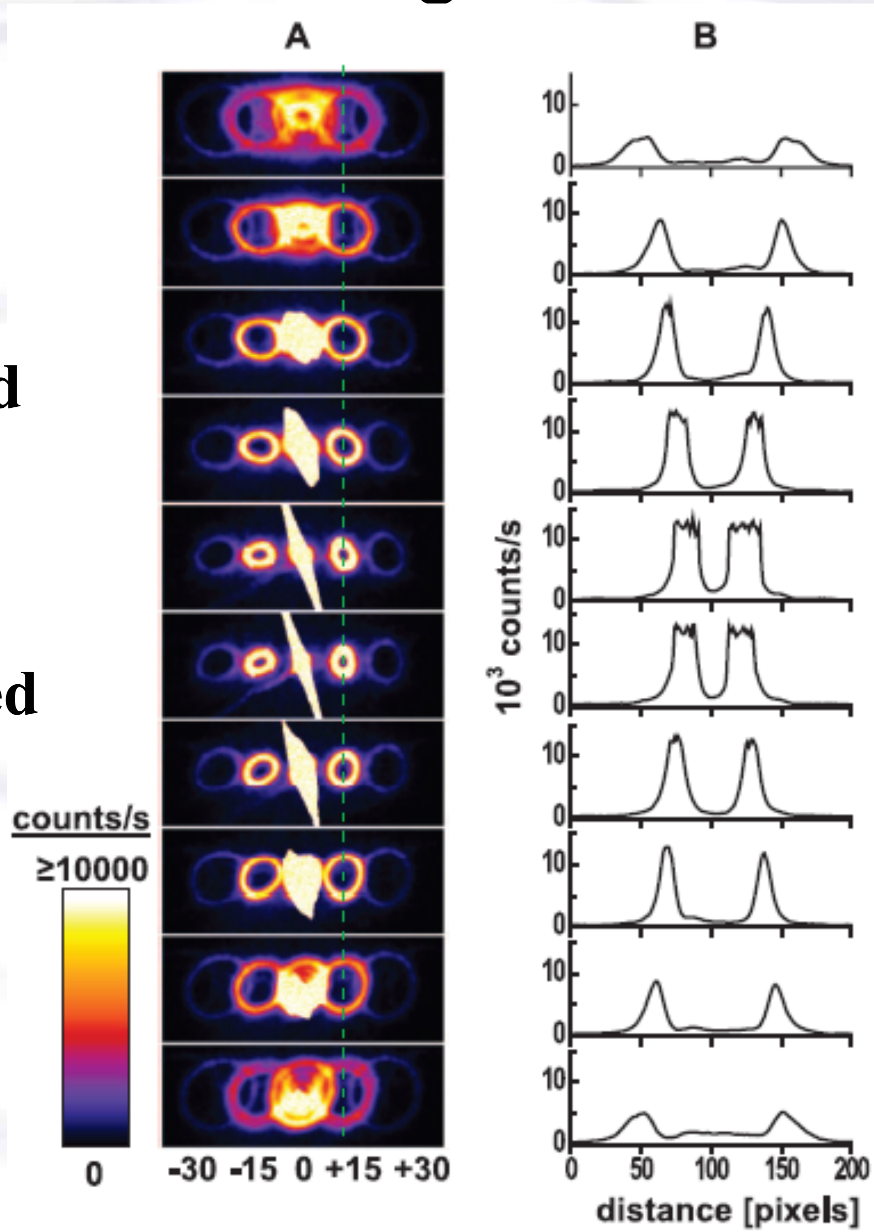
B J McMorran et al. Science 2011;331:192-195

Large Orbital Angular Momentum

Over-focused

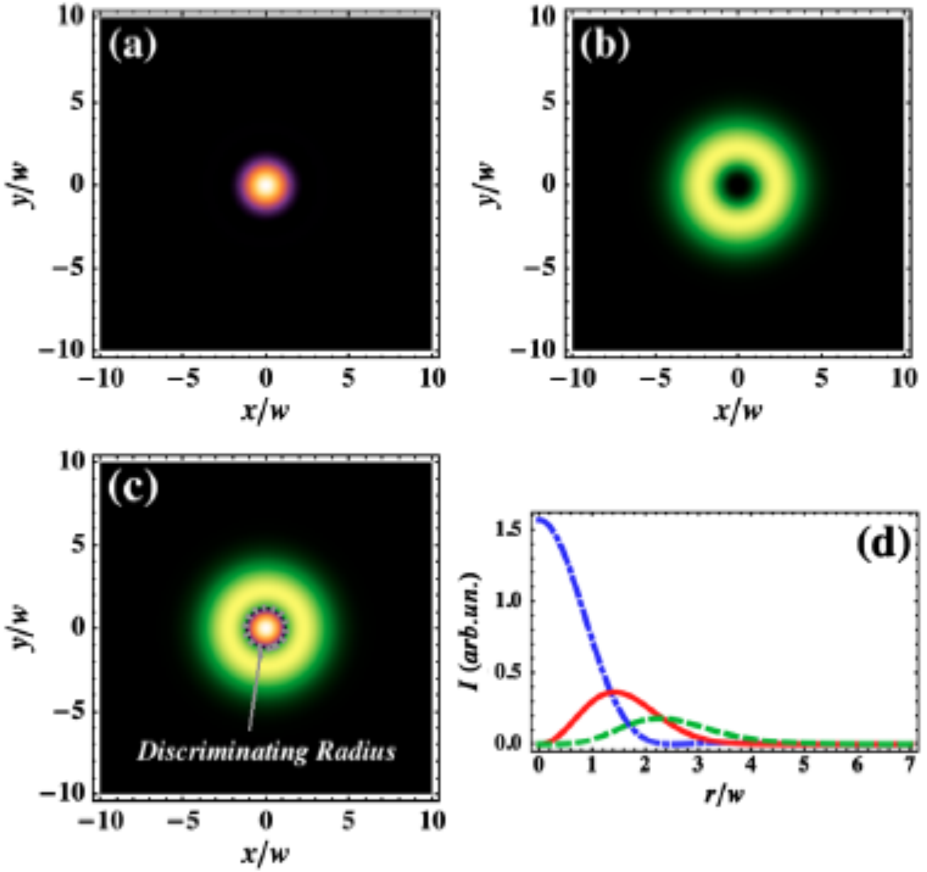
to

Under-focused



B J McMorran et al. Science 2011;331:192-195

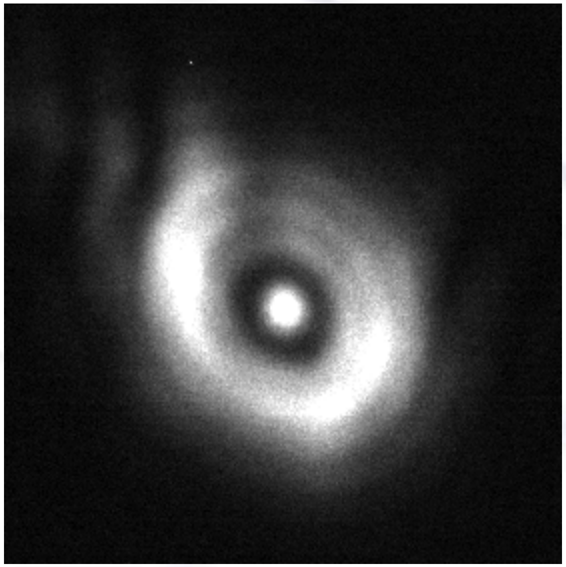
Spin Polarized Electron Beams

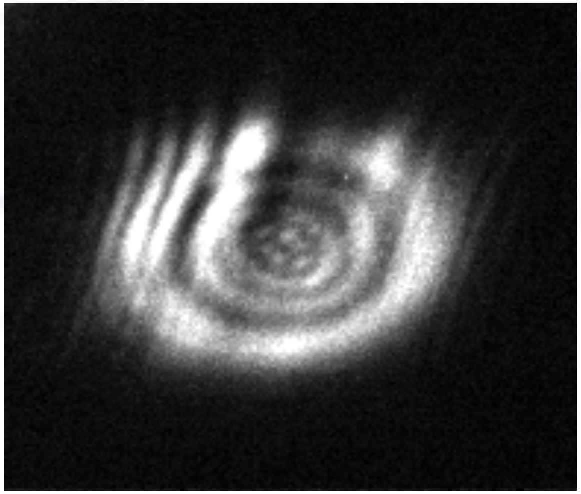


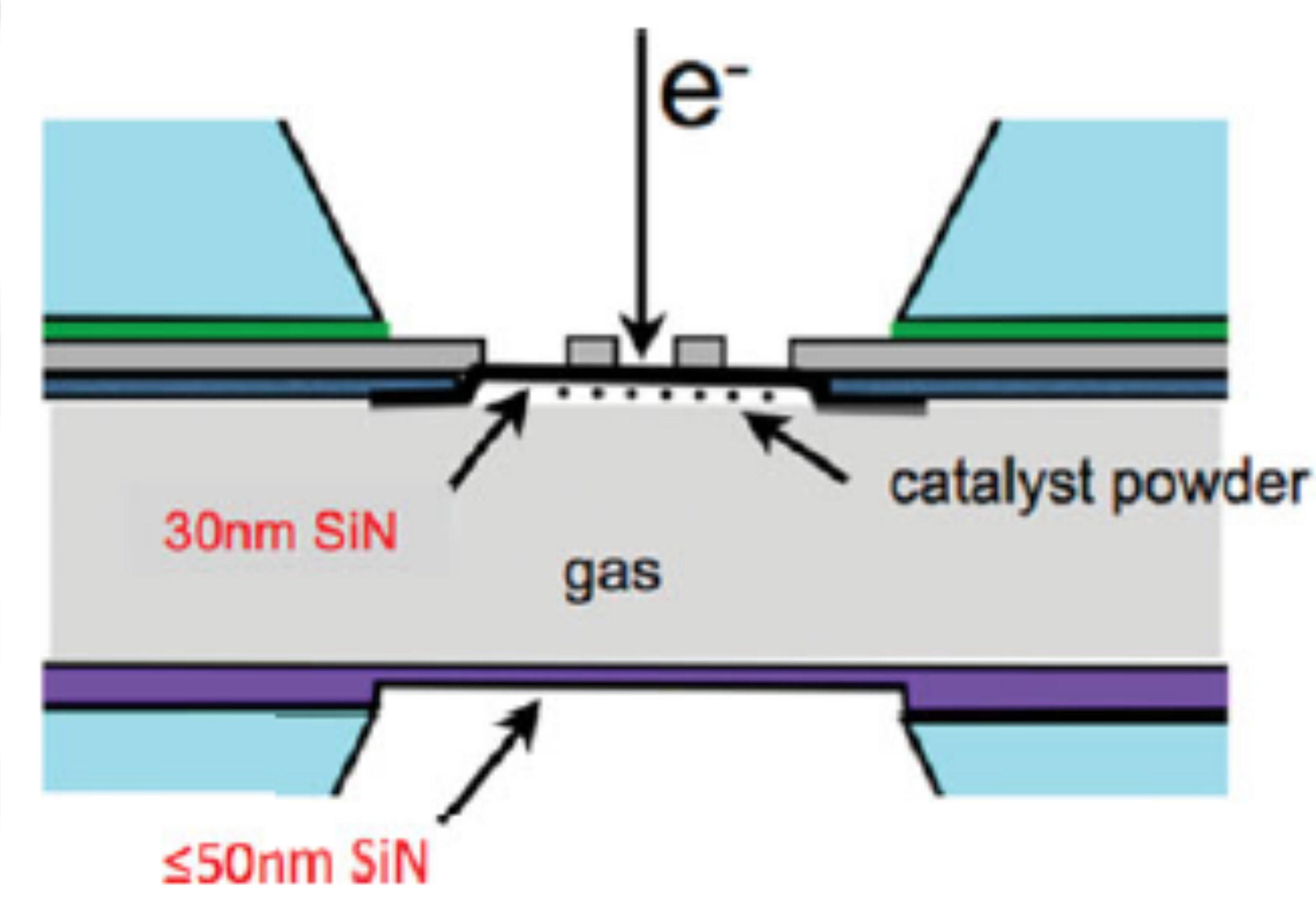
Far-field ExB-filtered beam profiles for a) l=0, b) l=2, c) together showing discriminating radius and d) intensity profiles.

Grillo et al, PRL 108 (2012) 044801.

STEHM's EVB + ExB images

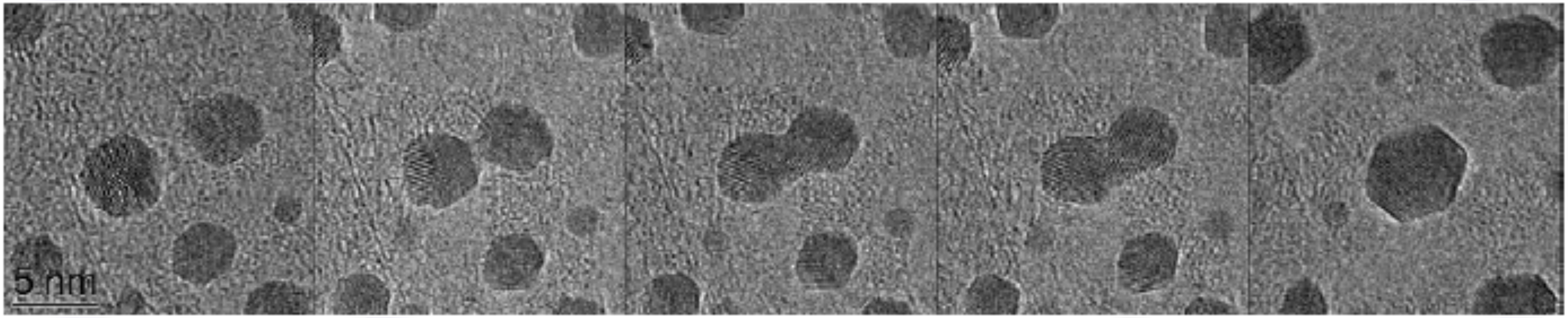






Specimens can be solids, liquids and gases.

Catalysts Interactions



Summary

The STEHM

- One awesome electron microscope

- Enabling awesome science

STEHM Team

Collaboration between:

Hitachi, HHT Japan + HHT Canada

CEOS, Germany

UVic, Canada

Funding Sources

- **Canadian Foundation for Innovation, CFI**
- **British Columbia Knowledge Development Fund, BCKDF**
- **Hitachi High Technologies Canada**
- **University of Victoria**

Acknowledgements

Contributions from the following individuals are greatly appreciated:

Elaine Humphrey, UVic

Adam Schuetze, UVic

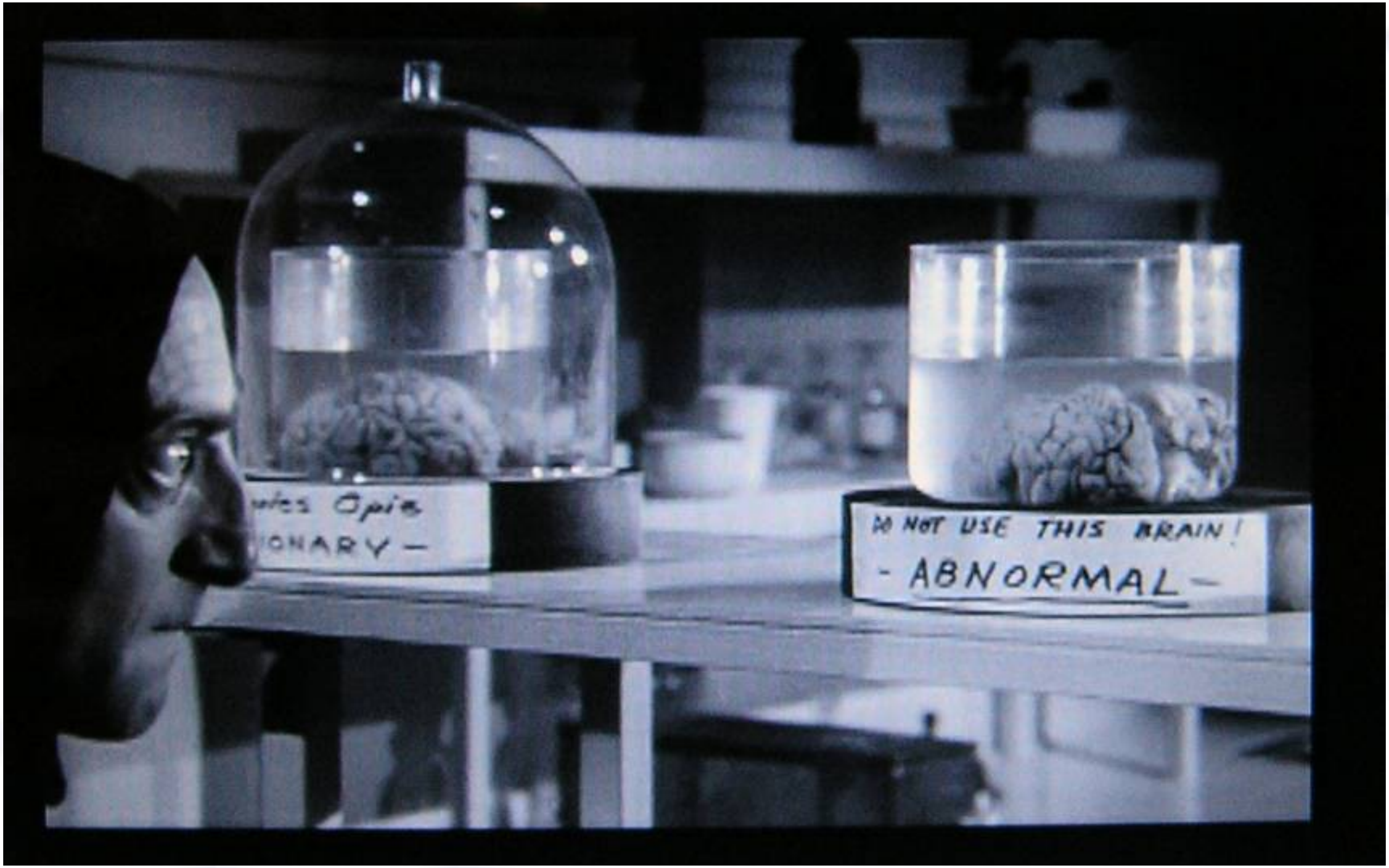
David Hoyle, HHT Canada

Acknowledgements

Communications with the following are greatly appreciated:

Giulio Pozzi
Archie Howie
Peter Schattschneider
John Spence
Ray Egerton
Harold Rose
Hannes Lichte
Takayoshi Tanji
Ben McMorran
Koh Saitoh
Karen Kavanagh

In Conclusion



In Conclusion

Don’t use the ABNORMAL infrastructure

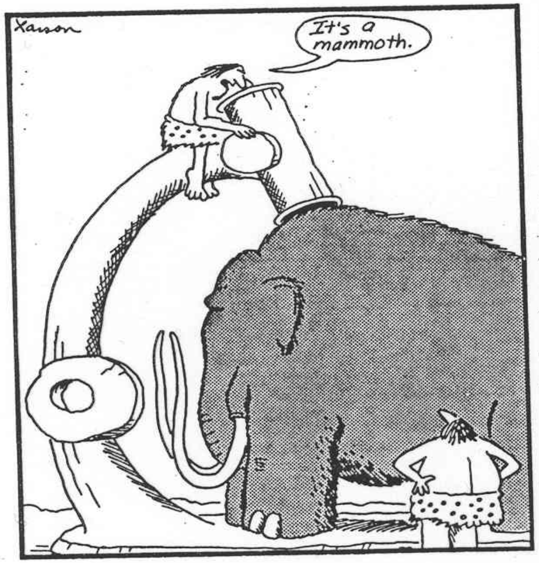
Use the STEHM infrastructure for what its worth!

See:

Lab Manager - Elaine Humphrey

Trainer and Engineer – Adam Schuetze

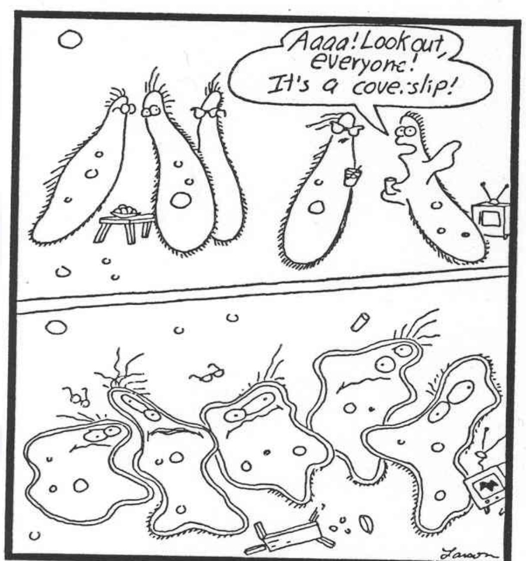
http://stehm.uvic.ca/



Early microscope

THE FAR SIDE By GARY LARSON

*Young urban scientist



Life on a microscope slide

NOVEMBE

30

THURSDAY